A HYBRID SYSTEM FOR THE TREATMENT OF OILFIELD PRODUCED WATER

BY

WALEED KHALID ALNOWAISER

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This thesis, written by **Waleed Khalid Alnowaiser** under the direction his thesis advisor and approved by his thesis committee, has been presented and accepted by the Dean of Graduate Studies, in partial fulfillment of the requirements for the degree of **MASTER OF SCIENCE IN CIVIL ENGINEERING.**

Dr. Muhammad H. Al-Malack
(Advisor)

Dr. Salah U. Al-Dulaijan
Department Chairman

Dr. Alaadin A. Bukhari
(Co-Advisor)

Dr. Salam A. Zummo
Dean of Graduate Studies

Dr. Muhammad S. Vohra
(Member)

26/5/16
Date

Dr. Basim Abussaud
(Member)

Dr. Alexis Nzila
(Member)

To My Family & Friends.

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LIST OF ABBREVIATIONS

PW: Produced Water

COD: Chemical Oxygen Demand

TOC: Total Organic Carbon

MLSS: Mixed Liquor Suspended Solids

MLVSS: Mixed Liquor Volatile Suspended Solids

MBR: Membrane Bioreactor

MSBR: Membrane Submerged Bioreactor

NORM: Naturally Occurring Radioactive Material

BOD: Biological Oxygen Demand

TDS: Total Dissolved Solids

TSS: Total Suspended Solids

DSA: Dimensionally Stable Anode

MF: Microfiltration

NF: Nanofiltration

UF: Ultrafiltration

RO: Reverse Osmosis

ED: Electro Dialysis

EDI: Electro Deionization

PAN: Polyacrylonitrile

PVDF: Polyvinylidene Difluoride

PES: Polyethylsulphone

PE: polyethylene

PP: polypropylene

SMP: Soluble Microbial Products

EPS: Extracellular Polymeric Substances

OC: Organoclay

CSTR: Completely Stirred Tank Reactor

DAF: Dissolved Air Flotation

SRT: Sludge Retention Time

HRT: Hydraulic Retention Time

ABSTRACT

Full Name : Waleed Khalid Alnowaiser

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In this research the organic pollutant removal performance and the mixed liquor characteristics of a hybrid system consisting of an electrocoagulation unit and a membrane bioreactor employing a moderately halophilic bacterial consortium for the treatment hypersaline produced water containing an oil content of 100, 150 and 200 mg/L were studied. An increase in both the MLSS and the MLVSS occurred in all the three stages. The bacterial colony plate count was used as a direct measure of the bacterial growth in the mixed liquor. The transmembrane pressure during each phase of the hybrid system remained in the range of 0.5 to 4.0 psi and the need for membrane washing never arose. The hybrid system showed a fairly good removal efficiencies with COD removal efficiency in the range of 92 – 96% and oil & grease removal efficiency in the range of 83 – 94%.

ملخص الرسالة

الاسم الكامل: وليد بن خالد بن رشيد النويصر

عنوان الرسالة: نظام هجين لمعالجة مياه آبار النفط

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في هذه الرسالة تمت دراسة قدرة المعالجة بالإضافة إلى خصائص النظام لنظام هجين يتكون من وحدة معالجة الكتروكيميائية بالإضافة إلى خزان يحتوي على فلتر عالي الجودة مغموس في بيئة عالية التركيز بالملوحة والبكتيريا لمعالجة مياه آبار النفط. تحتوي المياه المستخدمة في هذه الدراسة على نفط ثقيل بنسبة تتراوح بين 100 إلى 200 مليجرام لكل لتر مياه بحر. في كل الثلاث مراحل للدراسة كانت هناك زيادة ملحوظة في تركيز المواد المعلقة وتركيز المواد المعلقة الطيارة لخزان المعالجة. تم استخدام طريقة الحساب الكلي للبكتيريا لدراسة النمو البكتيري في النظام الهجين. على مدار الدراسة لم يتجاوز ضغط الفلتر عالي الجودة المستخدم في النظام الهجين حاجز الضغط المسموح لتشغيل الجهاز وبالتالي لم تظهر الحاجة للغسل العكسي للفلتر. أظهر النظام الهجين قدرة إزالة عالية لمعدل استهلاك الأكسجين الكيميائي ولعكارة المياه ومعدل الزيت.

CHAPTER 1

INTRODUCTION

Like most industrial activities, oil production processes generate large volumes of liquid waste that is called produced water (PW). Apart from its large generated volumes, PW has a diversified composition and contains different substances such as oil, salts, heavy metals, organic acids and radionuclides; therefore, PW is considered as a major source of contamination in oil fields [1]. Produced water has physical and chemical properties that vary considerably depending on the geographic location and formation of the field and the type of hydrocarbons being produced [2]. Due to the increase in the worldwide energy consumption, oil production has increased dramatically in the past decades, which led to a huge increase in volumes of produced PW. In 2007, Ferro and Smith estimated the global production of PW at around 250 million barrels per day compared with around 80 million barrels per day of produced oil [3], which clearly indicates a water to oil ratio of about 3:1. Moreover, Fakhru'l-Razi et al. estimated that more than 300 million barrels of PW will be produced in 2015 [2], accordingly, Saudi Arabia, the world's largest oil producer, will have to manage around 30 million barrels of PW each day. In addition to its large generated volumes, PW is considered as a strong wastewater in terms of contaminants concentrations, where oil & grease (O&G) and chemical oxygen demand (COD) concentrations of 1565 and 120000 mg/L, respectively, were reported [4,5]. Moreover, PW was reported to contain high concentrations of different heavy metals. Consequently, discharging PW

without proper treatment will definitely result in polluting receiving environments such as surface and underground water and soil.

Produced water is a mixture of organic and inorganic materials. Many factors affect the physical and chemical properties of produced water such as the geological location, the geological formation, the lifetime of the field and the operational conditions. Due to the different nature of oilfield reservoirs around the world it is difficult to but a small range of values for the different parameters and characteristics of produced water but mainly the major compounds of produced water are dissolved and dispersed oil compounds, dissolved minerals, dissolved gases and chemicals and solids used in the production processes. Discharging produced water can pollute surface and underground water and soil. The results and effects of discharging this huge amounts of produced water on the environment has recently become a significant issue of environmental concern. Moreover, countries with limited fresh water resources are progressively concentrating on efforts and researches to find efficient and cost effective methods to treat produced water.

Treatment of produced water is an effective option for produced water handling. Treatment of produced water convert it to be a valuable product rather than a waste. Nowadays, many single and joint physical, chemical, and biological methods are used for produced water treatment all around the world. Each one of these treatments methods have its advantages and disadvantages, thus a combination of two or more methods is always the case hence each of the stages will fulfill the other to reach the highest treatment levels. Selecting the best combination of methods is based on many factors such as the characteristics of the produced water, the space availability and the reuse and discharge plans. Eventually, a unique method or treatment system cannot be used with any produced water from any

oilfield around the world. Every year, the governments and the environmental agencies implement new and strict standards for discharging treated produced water to the environment. Newly developed hybrid systems can help to improve the final treatment effluent to meet the strict requirements.

This study will investigate the treatment of synthetic wastewater with the characteristics of a typical oilfield produced water. A comprehensive literature review carried out revealed that a combination of two treatment techniques in a hybrid system to treat oilfield produced water is a new area of research.

CHAPTER 2

LITERATURE REVIEW

2.1 Produced Water Generation and Characteristics

Produced water is water trapped in underground formations that is brought to the surface during oil and gas exploration and production. In traditional oil and gas wells, produced water is brought to the surface along with oil or gas. Because the water has been in contact with the hydrocarbon-bearing formation for centuries, it has some of the chemical characteristics of the formation and the hydrocarbon itself. It may include water from the reservoir, water injected into the formation, and any chemicals added during the drilling, production, and treatment processes. [1]

The physical and chemical properties of produced water vary considerably depending on the geographic location of the field, the geological formation from which it comes, and the type of hydrocarbon product being produced. Produced water properties and volume can even vary throughout the lifetime of a reservoir. [1]

The major constituents of interest in produced water are:

- Salt content: Salt content can be expressed as salinity, total dissolved solids, or electrical conductivity. The salt content in produced water varies widely, from nearly freshwater to salt levels up to ten times higher than seawater.

- Oil and grease: Oil and grease is not an individual chemical. Rather, the term "oil and grease" refers to a common test method that measures many types of organic chemicals that collectively lend an "oily" property to the water.
- Various inorganic and organic chemicals: These chemicals are found naturally in the formation, are transferred to the water through long-term contact with the hydrocarbon, or are chemical additives used during drilling and operation of the well. The presence of specific chemicals and the concentrations of those chemicals vary widely among different produced water samples.
- Naturally occurring radioactive material (NORM): Some of the formations holding oil and gas have small concentrations of natural radioactivity. Low levels of the radioactivity can be transferred into produced water. Generally, the radiation levels in produced water are very low and pose no risk. However, scale from pipes and sludge from tanks holding produced water can concentrate NORM.

Characteristics of different produced water from different literatures are showed in Table1.

Produced water is by far the largest volume byproduct stream associated with oil and gas exploration and production. Today, the global produced water production is estimated at around 250 million barrels per day [2]. The cost of handling this large amount of produced water is too high. According to Schlumberger, a worldwide oil production company, the produced water handling costs range from 5 to more than 50 cents per barrel of produced water [2].

The cost of managing produced water is a significant factor in the profitability of oil and gas production. The total cost (ranging from 5 to more than 50 cents per barrel) includes:

- The cost of constructing treatment and disposal facilities, including equipment acquisitions;
- The cost of operating those facilities, including chemical additives and utilities;
- The cost of managing any residuals or byproducts resulting from the treatment of produced water;
- Permitting, monitoring, and reporting costs; and
- Transportation costs.

Moreover, discharging produced water can pollute surface and underground water and soil. The results and effects of discharging this huge amounts of produced water on the environment has recently become a significant issue of environmental concern. Countries with limited fresh water resources are progressively concentrating on efforts and researches to find efficient and cost effective methods to treat produced water.

Treatment of produced water is an effective option for produced water handling. Treatment of produced water convert it to be a valuable product rather than a waste. Nowadays, many single and combined physical, chemical, and biological methods are used for produced water treatment all around the world. Each one of these treatments methods have its advantages and disadvantages. Selecting the best method is based on many factors such as the characteristics of the produced water, the space availability and the reuse and discharge plans. Eventually, a unique method or treatment system cannot be used with any produced water from any oilfield around the world. Every year, the governments and the environmental agencies implement new and strict standards for discharging treated produced water to the environment. Newly developed hybrid systems can help to improve the final treatment effluent to meet the strict requirements.

Table 2.1 A Full Comparison between Produced Water Characteristics from the Literature and ABQIQ GOSP Produced Water

Parameter/Heavy Metal (mg/L unless stated)	ABQIQ GOSP	Alley et al. [6]	Horner et al. [7]	Cakmakci et al. [8]	Tellez et al. [9]	Ebrahimi et al. [10]	Mondal and Wickramasinghe [11]	Tibbetts et al. [12]	Neff [13]
pH (no unit)	7.1	5.18 – 8.9	7.2 – 8.5	7.1 – 7.87	7.3 ± 0.2	6 – 7.5	8.41 – 8.70	4.3 – 10	
Alkalinity		300 – 380	300 – 380	nd					
Nitrate		1 – 2							
Sulfate		8 – 13686		355 – 390					≤ 1.0 - 8000
Oil & Grease	47	nd - 92	103.8	472 – 1565	147 ± 35			0.275	
TDS	40.8		704 – 1370		35023 ± 75		588 – 2090	5 – 800	
TSS	74.33		3.2 – 26	132 – 35830	85 ± 12			1.2 – 1000	
BOD			4.4 – 9.4	nd – 7000					
Hardness			5 – 20						
Conductivity (µS/cm)				14322 – 47600		162 – 213	1175 – 4190		
Phenols	0.0096			0.59 – 10				0.009 – 23	0.6 - 23
TOC						23 – 94	47.7 – 136.4		≤ 0.1 - >11000

Dissolved Oxygen				< 1.0					
COD	2800			588 – 24500	431 ± 25			1220	
Total Petroleum Hydrocarbons					126 ± 30				
Aluminum		0 – 0.06	nd						
Arsenic		0.17 – 0.857							0.000004 - 0.32
Barium		0.07 – 7.4						1.3 – 650	≤0.001 - 2000
Cadmium	<0.0005	0.03 – 0.2	nd	0.001 - <0.15				<0.005 – 0.2	0.0000005 - 0.49
Calcium		4 – 52920	2.5 – 300				1.7 – 11.0	13 – 25800	
Chloride			5 – 48						
Chlorine		36 – 238534							
Chromium		0.1 – 1.0	nd					0.02 – 1.1	≤ 0.000001 - 0.39
Cobalt	0.00061 – 0.00063			nd					

Copper	0.00316 – 0.00327	0.33 – 2.68	Nd	0.002 – 0.98				<0.002 – 1.5	≤ 0.000001 - 55
Iron	0.00988 – 0.01054	0.5	nd – 171	0.25 – 30				<0.1 – 100	≤ 0.0001 - 465
Lead	<0.0005		nd	0.003 – 0.52				0.002 – 8.8	≤ 0.000001 - 18
Manganese	0.00215 – 0.00225	1.4 – 8.1						<0.004 – 175	0.0002 - 7.0
Magnesium		2 – 5096	1.05 – 8.7				0.01 – 1.3	8 – 6000	
Mercury								<0.001 – 0.002	≤ 0.000001 - 0.075
Nickel	0.00501 – 0.00512	2.7 – 9.5	nd – 9.5	nd – 0.004					≤ 0.000001 - 1.67
Potassium		1.6 – 42.6	1.6 – 42.6				1.2 – 10.5	24 – 4300	
Silicon			1.4 – 13.2				7.4 – 14.4		
Sodium		405 – 126755	8.8 – 430	3165 – 18900			250 – 782	132 – 97000	
Strontium		0.05 – 2.2						0.02 – 1000	
Zinc	0.04202 – 0.04339	6.3 – 17.4	nd – 17.4	0.001 – 2.22				0.01 – 35	0.05 - 200

2.2 Treatment Techniques

Produced water is the aqueous liquid phase that is co-produced from a producing well along with the oil and/or gas phases during normal production operations. This includes water naturally occurring alongside hydrocarbon deposits, as well as water injected into the ground. The following are the main contaminants of concern in produced water:

- High level of total dissolved solids (TDS)
- Oil and grease
- Suspended solids
- Dispersed oil
- Dissolved and volatile organic compounds
- Heavy metals
- Radionuclides
- Dissolved gases and bacteria.
- Chemicals (additives) used in production such as biocides, scale and corrosion inhibitors, and emulsion and reverse-emulsion breakers

The amount of produced water, and the contaminants and their concentrations present in produced water usually vary significantly over the lifetime of a field. Early on, the water generation rate can be a very small fraction of the oil production rate, but it can increase with time to tens of times the rate of oil produced. In terms of composition, the changes are complex and site-specific because they are a function of the geological formation, the oil and water (both in-situ and injected) chemistry, rock/fluid interactions, the type of production, and required additives for oil-production-related activities.

Treatment has three main stages. In pre-treatment, the bulk of the oil and gas, as well as coarse particles are removed. This is followed by the main treatment, which focuses on further removal of small hydrocarbon droplets and small particles from the water. This is achieved in two steps of treatment. The primary step removes larger hydrocarbon droplets and large solid particles, as well as hydrocarbon slugs. A secondary step goes after smaller droplets and particles, and it encompasses the bulk of the de-oiling equipment used in the upstream industry. This secondary step is usually sufficient to reduce the dispersed hydrocarbon content to below the typical offshore discharge level of 40 mg/L. Then, there is a final polishing treatment, which can be optional, where the oil concentration is lowered to levels typically below 10 mg/L. The implementation of this last treatment depends on either the regulatory framework or an operational requirement if water is going to be re-injected for either disposal or as part of the water flood operations in the field. Sometimes an additional treatment is required, where the final effluent stream must have a high quality. In this case, oil concentration is typically below 5 mg/L, and there are usually other restrictions that must be met such as heavy metals content, chemical oxygen demand (COD) levels, and so on.

Considering the main contaminants present in produced water, treatment goals include de-oiling, desalination, degassing, suspended solids removal, organic compounds removal, heavy metal and radionuclides removal, and disinfection. These treatment goals are essentially the same for potable, non-potable reuse, or disposal, although the level of contaminant removal required for potable reuse can be significantly higher, depending on the quality of the produced water.

Achieving the various treatment goals requires the use of multiple treatment technologies, including physical, chemical, and biological treatment processes [2]. Some of the technologies employed are:

Removal of dissolved organic compounds:

1. Adsorption;
2. Dissolved air precipitation;
3. Chemical (e.g., ozonation, fenton process), electrochemical or photocatalytic oxidation;
4. Biological degredation; and
5. Nano filtration or reverse osmosis.

Removal of metals:

1. Aeration + settling + sand filtration;
2. Ion exchange; [3] reverse osmosis.

Suspended solids removal:

1. Coagulation/flocculation + sedimentation + filtration;
2. Microfiltration or ultrafiltration.

Research on treatment strategies for reclamation of domestic and industrial wastewater for reuse requiring high water quality has centered on membrane treatment [3]. Membranes can remove TDS and a wide range of dissolved organic compounds at very high efficiency. The main problem with the membrane treatment is the high TDS and oil contents of produced water, which result in very high osmotic pressure and fouling of the membranes by hydrocarbon compounds. The pretreatment processes remove oil droplets, suspended

solids (e.g., coagulation and flocculation, sand or membrane filtration), metals (e.g., chemical precipitation), and dissolved organic compounds (e.g., adsorption, chemical oxidation, and biological degradation), and protect the membranes from excessive fouling at the same time. Many oil fields already have extensive treatment facilities for discharge requirement. These existing treatment processes can be used as the pretreatment for membrane treatment. At locations where land is more available, biological treatment could be a low-cost option for membranes pretreatment. Pretreatment or post treatment processes are also needed to remove dissolved gas and uncharged, low-molecular weight compounds, which are not removed by membranes [5].

2.3 Electrocoagulation

Electrocoagulation is an innovative approach for treating produced water and as a pre-treatment to membrane treatment for emulsified oils, heavy metals and other constituents in the oil and gas industry. Electrocoagulation is an electrochemical method where coagulants are produced in-situ by passing D.C. current through aqueous media. Sacrificial anodes are dissolved in order to produce the coagulants. In addition, hydrogen gas is evolved from cathode and oxygen or chlorine gas may evolve from anode. The electrodes are generally made of aluminum or iron. Briefly, EC is a hybridization of coagulation, floatation and electrochemistry [4]. Electrocoagulation (EC) generally refers to a group of technologies which use an electrical current that coagulates organic constituents and suspended solids in water. The coagulated organics have the ability to adsorb certain ionic constituents, making it possible to separate a flocculent with a majority of the suspended organics and some of the ionic constituents removed. Another variant of this system

oxidizes an iron or aluminum anode to form an iron or aluminum hydroxide flocculent which can co-adsorb/co-precipitate some ions. Multiple mechanisms have been claimed for removal of contaminants from water.

EC is an active process that involves three major parts:

1. The reaction chamber containing single or multiple anode and cathode pairs through which contaminated water passes. The electrodes can be designed as plates, perforated plates, or tubes. They can be composed of different materials, including aluminum, iron, stainless steel, and titanium. A series of reaction chambers can be used, each with different electrode material.
2. The electrical system, composed of control electronics. The current passed to the electrodes is often designed to be alternating (AC). Typically direct current (DC) is required, although using alternating current (AC) technology may prevent formation of an oxide layer on the cathode.
3. A system to dewater the precipitated/coagulated solids. This system could be similar to any used in conventional chemical precipitation processes.

Electrocoagulation may have certain niche applications where the technology may be effective, including near-neutral waters where co-precipitation with iron hydroxide could polish relatively clean waters. Potential applications include the following:

- Final treatment and polishing of discharge water from a high-density sludge water treatment plant to remove residual colloidal material and metals.
- Pre-treating water prior to Pressure-Driven Membrane Separation to remove colloidal silica and metals near saturation.

- Treating neutral tailings water to remove minor amounts of metals prior to discharge (generally will not be successful treating total dissolved solids (TDS) or sulfate in this type of water)

Prior testing of these applications must be conducted to verify the performance with each water type.

Electrocoagulation is an alternative to chemical precipitation for the removal of dissolved and suspended metals in aqueous solutions. The quantity of sludge produced is lower. The flocs generated are larger and heavier and settles out better than in conventional chemical precipitation processes. Since a large thickener is not required, capital costs can also be lower. The effluent generated by electrocoagulation contains no added chemicals and is often of better quality, containing TDS and less colloidal particulates. Reduction of TDS has been reported at 27%–60%, and reduction of total suspended solids can be as great as 95%–99% [5]. Although electrocoagulation requires energy input, it requires only low currents and can be operated using green technologies such as solar or wind power. [6]

The EC process destabilizes suspended, emulsified or dissolved contaminants in an aqueous medium by introducing an electrical current into the medium. The electrical current provides the electromotive force to drive the chemical reactions. When reactions are driven or forced, the elements or compounds will approach the most stable state. Generally, this state of stability produces a solid that is either less colloidal or less emulsified (or soluble) than the compound at equilibrium values. As this occurs, the contaminants form hydrophobic entities precipitate and can easily be removed by a number of secondary separation techniques.

Electrocoagulation utilizes direct current to cause sacrificial electrode ions to remove undesirable contaminants either by chemical reaction/oxidation and precipitation, or by causing colloidal materials to coalesce and then be removed by electrolytic flotation.

The electrochemical reactions may be summarized as follows:

At the anode: $M(s) \rightarrow M3+ (aq) + 3e-$ (1)

At the cathode: $3H2O(l) + 3e- \rightarrow 32H2 + 3OH-$ (2)

In the solution: $M3+ (aq) + 3H2O \rightarrow M(OH3)(s) + 3H+ (aq)$ (3)

M3+ (aq) and OH− ions generated by the electrode reactions (1) and (2) react, respectively, to form various monomeric species, depending on pH range, which transform finally into M(OH)3 according to complex precipitation kinetics. Freshly formed amorphous M(OH)3 (sweep flocs) with large surface areas which are beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles. Consequently, these flocs can be removed by sedimentation or by flotation using H2 bubbles produced at the cathode [7].

In general, there are various parameters which have an effect on the efficiency of the EC in removing the pollutants from water. Parameters which are known to have an effect are:

- Material of the electrodes can be iron, aluminum and/or inert material (typically cathodes). Iron and aluminum ions and hydroxides have different chemistries and applications.

- pH of the solution has an effect on the speciation of metal hydroxides in the solution and also on the potential of the colloidal particles. It also affects the dissolution of aluminum cathodes.
- Current density is proportional to the amount of electrochemical reactions taking place on the electrode surface.
- Treatment time or electric charge added per volume is proportional to the amount of coagulants produced in the EC system and other reactions taking place in the system.
- Electrode potential defines which reactions occur on the electrode surface.
- Concentration of the pollutants affects the removal efficiency because coagulation does not follow zeroth-order reaction kinetics but rather pseudo second or first-order kinetics.
- Concentration of anions, such as sulphate or fluoride, affects the composition of hydroxides because they can replace hydroxide ions in the precipitates.
- Temperature affects floc formation, reaction rates and conductivity. Depending on the pollutant, increasing temperature can have a negative or a positive effect on removal efficiency.
- Other parameters, such as hydro dynamical conditions and inter-electrode distance, may have effect on efficiency of the treatment and electricity consumption. However, these parameters have not been widely studied or they have a negligible effect on the removal efficiency.

The electrocoagulation process is complex. No set configuration is applicable to all needs, and many parameters need to be adjusted for optimal treatment.

Zhao et al. [8] studied hardness removal (together with chemical oxygen demand (COD) and turbidity) from the produced water by a pilot-scale electrocoagulation (EC) system to mitigate the scaling and fouling of Reverse Osmosis (RO) membranes. This was critical for mitigating membrane scaling and fouling. A preliminary research was employed to obtain desired operating conditions: initial pH 7, current density 5.56 mA/cm2 and electrolysis time 30 min. RSM with an eighteen-run CCD design was performed and second-order regression models were generated. ANOVA was conducted to validate the significant consistency between experimental values and predicted ones (hardness removal: R2 0.9782, F value 39.80, p-value b0.0001; COD removal: R2 0.9752, F value 34.97, p-value b0.0001; turbidity removal: R2 0.9865, F value 65.15, p-value b0.0001). The pH and current density had the most significant effects on pollutant recovery, while reaction time was least significant. The maximized hardness, COD, and turbidity removal efficiencies were 85.81, 66.64, and 93.80%, respectively. The final steady flux was around 22 L/(m2 h) and the water recovery rate reached 87.83%. These indicated that RSM was an effective approach for obtaining desired operating conditions in complex EC pretreatment processes for RO membrane reactors.

Tir and Moulai-Mostefa [7] studied Optimization of oil removal from oily wastewater by electrocoagulation using response surface method. In this study, the efficiency of electrocoagulation process applied to the treatment of an oily wastewater emulsion was investigated. It was shown that electrocoagulation treatment achieves a fast and effective removal of turbidity and chemical oxygen demand. The treatment efficiency was found to be function of the initial pH, applied current density and electrolysis time. The microscopic analysis confirmed the destabilization of the emulsion by the coagulant (Al3+) coming

from the dissolution of aluminium anode, with the formation of large flocs size. The quadratic model developed in this study shows the presence of a high correlation between experimental and predicted values. Analysis of variance showed a high coefficient of determination value (R2 = 0.998), thus ensuring a satisfactory adjustment of the second-order regression model with the experimental data. Under optimal values of process parameters (current density = 25 mA/cm2, initial pH 7, electrolysis time = 22 min) more than 99% removal turbidity and 89.63% removal COD were obtained.

Santos et al. [9] studied the application of electrochemical technology to the remediation of oily wastewater. The successful application of electrochemical technology, employing a dimensionally stable anode (DSA), for the remediation of wastewater from the oil extraction industry has been demonstrated. It has been established that DSA electrodes can be very active in reducing the content of organic components present in oily wastewater samples. Voltammetric and chronoamperometric experiments revealed that the reduction of organic material occurred at the same potential as the OER (i.e. above 1.100 V). Except for the regular redox processes of Ru in the solid state, no electrochemical activity was detected in the potential range 0.200–1.100 V. At potentials higher than 1.100 V, the anodic displacement of the OER revealed a breakdown in acid–base equilibrium at the metal oxide layer because the thin film of adsorbed organic material inhibited access of water to the active layer thus preventing the formation of the necessary OH. The removal of COD was influenced by temperature, increases in which mainly favored the electrochemical process. At a current density of 100 mA/cm2, the most efficient COD reduction was achieved at 50^0C reaching 40% after 12 h, and 57% after 70 h. The reduction in COD could be attributed to electro-oxidation/electrode-gradation and also to electro-flotation processes.

Karhu et al. [10] studied Bench scale electrocoagulation of bio oil-in-water and synthetic oil-in-water emulsions. Bench scale EC test runs for bio and synthetic oil-in-water emulsions with SS and Al anodes were performed. The final pHs of treated waters of similar types were higher for a SS anode than for an Al anode. The pH increased more for the bio O/W emulsions than for synthetic O/W emulsions regardless of the anode material used. The precipitate produced by the SS anode was more easily separated than the Al precipitate, but contained more bound water. The test runs conducted succeeded very well with high reductions of COD, TOC, TSC and turbidity for both anode materials. It appears that the SS anode performed slightly better for synthetic O/W emulsions and the Al anode for bio O/W emulsions. Laser diffraction and BOD analyses conducted for waters of a number of test runs proved the breaking of O/W emulsions. The current behaved differently when using SS or Al as the anode material. It was assumed that the structure of the produced precipitate and the unusual structure of the EC cell were behind this. Energy consumption was low for both types of anodes. The dissolution of Fe2+ from the SS anode was calculated to be significantly higher than the Al3+ dissolution from the Al anode. Therefore, the treatment costs using SS as anode material were almost trebled compared to the treatment costs using Al. Analyses of the samples taken during test runs 8–16 revealed that the most likely optimal treatment times would have been much shorter than the total treatment times used here, even as short as 20 min for SFO and MO + MCO emulsions with Al as the anode material. The EC process using SS or Al as the anode material was found to be a cost-effective and feasible method for breaking the stable bio and synthetic oil-in-water emulsions. This is even without the specific optimization of the most important process parameters.

Emamjomeh and Sivakumar [11] reviewed the pollutants removed by electrocoagulation and electrocoagulation/flotation processes. The experimental results showed that aluminium electrodes are more efficient than iron electrodes. It may be because of high adsorption capacity of hydrous aluminium oxides for oil removal. The electrocoagulation technology is also considered to be potentially an effective tool for treatment of color from textile wastewaters with high removal efficiency. The removal efficiency was found to be dependent on the initial pH, the dye concentration, the applied current density, and the electrolysis time in batch model. It was found that maximum 98.5% of the dye was removed from the solution by EC technology. The capacity and efficiency of the EC process for treatment of industrial wastewaters was found to be dependent on the nature of the liquid waste and initial pollutant concentrations. The results on the investigation of the EC process for treatment of heavy metals indicated that using an EC reactor successfully removes arsenic and cadmium from water or wastewater. The highest removal of arsenic was obtained by using iron electrodes when the removal mechanism was considered to be adsorption of arsenic on hydrous ferric oxides. However, no evidence was reported to support it. Other research studies showed that EC process is more efficient than coagulation process for potable water treatment. Less aluminium dosage was needed to get the same removal efficiency in EC process, which is related to chemical cost and sludge production. The results on the investigation of the electrocoagulation process for treatment of nitrate, nitrite, and ammonia indicated that the pollutants can be removed from aqueous solution.

Esmaeilirad et al. [12] studied Influence of softening sequencing on electrocoagulation treatment of produced water. The impact of sequencing of softening with electro-coagulation treatment of produced water was examined. Overall, softening at a lower pH

(9.5) before EC was more effective than softening after EC with a higher pH (10.2). In particular, Soft-EC removal efficiency was greater for Ba, B, Sr, Ca, Mg, Fe, and TOC. Other observation include: Both treatment sequences showed similar solids removal effectiveness (as measured by turbidity) after one month or more of flow back. This is likely due to the decrease in organic matter constituents that have not been identified. However for the early flow back water (one-day and two-day old samples), there was not a substantial difference in suspended solids removal efficiency between Soft-EC and EC-Soft. Both EC sequences were ineffective coagulating and flocculating colloids resulting in unacceptable treatment.

2.4 Membrane Bioreactor Systems

Membrane bioreactor (MBR) is the combination of a membrane process like microfiltration or ultrafiltration with a suspended growth bioreactor, and is now widely used for municipal and industrial wastewater treatment [17]. The Membrane Bioreactor (MBR) process is an emerging advanced wastewater treatment technology that has been successfully applied at an ever increasing number of locations around the world. In addition to their steady increase in number, MBR installations are also increasing in terms of scale. This innovative technology offers several advantages over the conventionally activated sludge process. Among these advantages are higher biomass concentration, eliminating the needs of secondary clarifiers and improved effluent quality.

The use of MBR systems allows for higher wastewater flow or improved treatment performance in a smaller space than a conventional design, i.e., a facility using secondary clarifiers and sand filters. Historically, membranes have been used for smaller-flow

systems due to the high capital cost of the equipment and high operation and maintenance (O&M) costs. Today however, they are receiving increased use in larger systems. MBR systems are also well suited for some industrial and commercial applications. The high-quality effluent produced by MBRs makes them particularly applicable to reuse applications and for surface water discharge applications requiring extensive nutrient (nitrogen and phosphorus) removal.

The advantages of MBR systems over conventional biological systems also include better effluent quality, smaller space requirements, and ease of automation. Specifically, MBRs operate at higher volumetric loading rates which result in lower hydraulic retention times. The low retention times mean that less space is required compared to a conventional system. MBRs have often been operated with longer solids residence times (SRTs), which results in lower sludge production; but this is not a requirement, and more conventional SRTs have been used [18]. The effluent from MBRs contains low concentrations of bacteria, total suspended solids (TSS), chemical oxygen demand (COD), and biochemical oxygen demand (BOD). This facilitates high-level disinfection. Effluents are readily discharged to surface streams or can be sold for reuse, such as irrigation.

In MBR system, secondary clarifiers and tertiary filtration processes are eliminated, thereby reducing plant footprint. In certain instances, footprint can be further reduced because other process units such as digesters or UV disinfection can also be eliminated or minimized. Unlike secondary clarifiers, the quality of solids separation is not dependent on the mixed liquor suspended solids concentration or characteristics. Since elevated mixed liquor concentrations are possible, the aeration basin volume can be reduced, further reducing the plant footprint. No reliance upon achieving good sludge settle ability, hence

it is quite amenable to remote operation. It can be designed with long sludge age, hence low sludge production. It produces a MF/UF quality effluent suitable for reuse applications or as a high quality feed water source for Reverse Osmosis treatment. [19]

A detailed holistic cost comparison may reveal reasonably comparable results between the costs of the MBR option versus other advanced treatment options, especially if land value is considered. Furthermore, whilst the costs for conventional technologies are slowly rising with labor costs and inflationary pressures, the costs for all membrane equipment (both for direct filtration and MBR) has been falling steadily during each of the last 10 years. Hence on a capital cost basis for any given project, the likelihood of MBR becoming a favored option is increasing with time. Designers are therefore advised to continuously re-assess the cost information for their particular project as it progresses through the various planning stages over time. [13]

The membrane process is a very important separation process in water and wastewater technology, which becomes increasingly competitive and is superior to the traditional water technology with proven performance and process economics. The most widely applied membrane separation processes are microfiltration (MF), ultrafiltration (UF), Nano filtration (NF), reverse osmosis (RO), electro dialysis (ED) and electro deionization (EDI), whereas the first four processes produce permeate and concentrate. The separation ranges are as follows: 100 to 1000 nm for MF, 5 to 100 nm for UF, 1 to 5 nm for NF, and 0.1 to 1 nm for RO. Firstly, application of membrane-based technologies in wastewater treatment was focused on tertiary treatment of secondary effluent, so as to obtain a high-quality final effluent that can be reused for different purposes. However, over the past 10 years, MBRs

have emerged as an effective secondary treatment technology, whereas membranes applied are usually in the range of those of MF and UF.

Membranes are usually made from different plastic and ceramic materials, but metallic membranes also exist. The most widely used materials are celluloses, polyamides, polysulphone, charged polysulphone and other polymeric materials such as polyacrylonitrile (PAN), polyvinylidene difluoride (PVDF), polyethylsulphone (PES), polyethylene (PE), and polypropylene (PP). All of these polymeric materials have a desirable chemical and physical resistance. They are also hydrophobic, and it is known that hydrophobic membranes are more prone to fouling than hydrophilic ones due to the fact that most interactions between the membrane and the foulants are of hydrophobic nature (20). All commercially available membranes are therefore modified by chemical oxidation, organic chemical reaction, plasma treatment, or by grafting to achieve more hydrophilic surface. This modification process usually differs one membrane from another together with the method of fabrication of the membrane module.

Alzahrani and Mohammad [14] studied the challenges and trends in membrane technology implementation for produced water treatment. Membrane technology implementation in the petroleum industry is in its infancy. Current applications of membrane technologies have demonstrated their high potential for meeting the petroleum industry's needs and their feasibility for treating produced water. The major functions of membrane technology for produced water treatment are driven by environmental regulations and the requirement to meet strict reuse standards for beneficial applications and re-injection in disposal wells. The findings of this review indicate that despite the success of current membrane technology applications, several challenges persist, such as membrane fouling, which

occurs due to the complex contaminants in produced water, including organic matter, scale compounds, boron and silica, and the absence of standard cleaning procedures. Ongoing research to mitigate these challenges has suggested solutions, including sufficient pre-treatment steps, the modification and development of novel membrane materials, and the use of Nano-sized particles to construct membranes and optimize cleaning procedures. These solutions have optimized the performance the production of anti- fouling membranes and the development of new cleaning agents. The current trends in membrane technology applications in the petroleum industry indicate that the need for membrane technology will continue to grow, as contemporary produced water management practices have shifted their focus from discharge and re-injection to reducing, reusing, and recycling. Thus, oil producers and petroleum refineries are being compelled to change their strategies for produced water management, and much attention is shifting to membrane technologies.

Kose et al. [15] studied performance evaluation of a submerged membrane bioreactor for the treatment of brackish oil and natural gas field produced water. Membrane flux was maintained at 10L/m2/h with a constant aeration intensity during the 297days operation. Cleaning was conducted once when trans-membrane pressure (TMP) reached 800mbar. The corresponding permeability after physical cleaning was restored to 60% and to 95% after subsequent chemical cleaning. Therefore, the TMP increase was caused by both the physically reversible cake layer and irremovable (chemically reversible) fouling. Element analysis was also performed on the hollow fiber membrane surface layer in order to identify the chemical components of the layer by EDX analysis. In the lab-scale MBR, membranes exhibited inhomogeneous fouling, depending on the position of fibers and module in the filtration line and on the age of the membrane. The elements of C, O, Na, Mg, Al, Si, S, Cl,

K, Fe, and Ca were detected. The C element came from organic matter in the mixed liquor adsorbed on membrane surface. The other elements were due to some inorganic precipitates originating from produced water. After one-year operation of lab-scale MBR system for the treatment of produced water, a stable performance is obtained in spite of the variations in the influent at different sludge retention times (SRTs). Although the COD removal in the bioreactor slightly increased with shortened SRT, the total COD removal efficiency of the MBR process could be kept over 80–85% independent of SRT. The COD removal rate slightly increased with SRT due to the higher concentration of biomass which may decompose organic compounds. The increase of sludge age increased the removal efficiency of oil and grease dramatically from 60% to 85%. The hydrocarbons removal efficiency of 99% was achieved. The corresponding permeability after physical cleaning was restored to 60% and to 95% after subsequent chemical cleaning.

Johir et al. [16] studied effect of salt concentration on membrane bioreactor (MBR) performances. The increased of salt concentration showed an inhibitory effect on organic and ammonia removal efficiency. The DOC and NH4-N removal reduced from 77 and 93% to 10 and 0% when salt concentration reached 35 g-NaCl/L. The amount of DON present in biopolymers increased (from 0.05 to 3.31 mg/L) with higher salt concentrations. The TMP development at different salt concentrations of 0 to 35.0 g-NaCl/L was marginal at around 2.0–10.50 mbar. Organic (DOC) and DON removal pattern can be classified into three distinct clusters (i) 0–3.0, (ii) 5–15 and (iii) 20–35 g-NaCl. It was concluded that the MBR process could be useful to treat saline water under low salt concentration. Its performance can be further improved by acclimatizing for sufficient time or by acclimatizing halophiles microorganisms naturally.

Zhang et al. [17] studied treatment of produced water from polymer flooding in oil production by the combined method of hydrolysis acidification-dynamic membrane bioreactor–coagulation process. After treatment by combined hydrolysis acidification-DMBR-coagulation process, the final effluent of PWPF from Shengli Oilfield, China, can meet the class I of National Wastewater Discharge Standard of China (GB8978-1996). The average concentration of NH3–N and COD reached 2.04 mg/L and 52.25mg/L, respectively, and the concentration of residual oil was too low to be measured. The highest acidification efficiency of HAR was 10.98% under the optimal HRT of 12 h. The concentration of oil decreased greatly from 35.47mg/L to 5.98 mg/L with the HRT of 12h, while the removal efficiency of COD and HPAM were only 9.68% and 12.32%, respectively. The recovery stage of the DMBR was longer than 20h. The stable stage can last from 30 h to110 h. At the stable stage the average concentration of NH3–N in effluent from DMBR was 1.50 mg/L, and the concentration of oil could not be measured. The average concentration of COD was as high as 476.63 mg/L. Al2(SO4)3 was found to be the optimal coagulant in coagulation process to treat the effluent from DMBR, which had the highest COD removal efficiency. The removal efficiency of COD reached 89.41% with the dosage of 140 mg/L at pH of 9.0, and 88.37% with the dosage of 80mg/L at optimal pH of 5.0.

Fakhru'l-Razia et al. [18] studied application of membrane-coupled sequencing batch reactor for oilfield produced water recycle and beneficial re-use. A lab-scale crossflow membrane sequencing batch reactor inoculated with isolated tropical halophilic microorganisms was used effectively for oilfield produced water treatment. It was found that the isolated microorganisms played an important role in the biodegradation of the

pollutants and membrane separation was required for ensuring a stable permeate quality. It was thus feasible to treat the produced water using the MSBR and MSBR/ RO, and the product quality met the requirements for discharge and re-use.

Di Bella et al. [19] studied performance of a MBR pilot plant treating high strength wastewater subject to salinity increase: i.e. Analysis of biomass activity and fouling behavior. The short terms effects of a sharp salinity increase in a pilot plant MBR treating high strength wastewater were investigated. Treatment efficiency, biomass activity and membrane fouling were studied. Under normal salinity, the pilot plant showed high removal efficiencies and the high OUR values highlighted intense biomass activity. The increase of salt concentration caused a decrease of respiration rates; the autotrophic populations highly suffered the stress exerted by salinity increase. The main mechanism of fouling was the irreversible cake deposition on the membrane surface, likely due to the worsening of sludge characteristics and high EPS_{bound} concentration. A rapid changing in salinity severely affect the membrane fouling and biomass activity, suggesting to investigate the opportunity to acclimatize the biomass to a gradual and moderate salinity increase.

Sharghi and Bonakdarpour [20] studied organic removal efficiency and halophilic bacterial mixed liquor characteristics in a membrane bioreactor treating hypersaline produced water at varying organic loading rates. The organic pollutant removal performance and the mixed liquor characteristics of a membrane bioreactor (MBR), employing a halophilic bacterial consortium, for the treatment of hypersaline synthetic produced water – at varying organic loading rates (OLR) from 0.3 to 2.6 kg COD m^{-3} d^{-1}– were considered. The oil and grease (O&G) and COD removal efficiency were 95–99% and 83–93%, respectively with only

transient O&G (mainly polycyclic aromatic hydrocarbons) and soluble microbial products accumulation being observed. With increasing OLR, in the range 0.9–2.6 kg COD m^{-3} d^{-1}, as a result of change in both extracellular polymeric substances (EPS) and zeta potential, bio flocculating ability improved but the compressibility of the flocs decreased resulting in the occurrence of EPS bulking at the highest OLR studied. The latter resulted in a change in the rheology of the mixed liquor from Newtonian to non-Newtonian and the occurrence of significant membrane fouling. A MBR operated at OLRs up to 2.6 kg COD m^{-3} d^{-1} was considered for the treatment of synthetic hypersaline PW using a halophilic bacterial consortium and high O&G and fairly good COD removal efficiency was achieved. Only transient accumulation of both O&G and SMP was observed. Increase in OLR, mainly as a consequence of change in EPS and ZP, resulted in pronounced change in halophilic bacterial morphology. The occurrence of EPS bulking at the highest OLR resulted in change in the mixed liquor rheology to non-Newtonian and relatively high membrane fouling rates.

Sharghi et al. [21] studied treatment of hypersaline produced water employing a moderately halophilic bacterial consortium in a membrane bioreactor: Effect of salt concentration on organic removal performance, mixed liquor characteristics and membrane fouling. The organic pollutant removal performance and the mixed liquor characteristics of a membrane bioreactor (MBR), employing a moderately halophilic bacterial consortium, for the treatment of hypersaline synthetic produced water containing 100–250 g/L NaCl were considered. The COD and oil and grease (O&G) removal efficiencies in the range 81.6–94.6% and 84.8–94.0% respectively and MBR effluent turbidity lower than 2 NTU were achieved. There was no pronounced membrane fouling at any salt concentration. O&G

accumulation (less than 11% of the influent O&G) occurred in the mixed liquor at all salt concentrations, but biodegradation was identified as the major organic removal mechanism. With increasing salt concentration, initially increase in SVI and later formation of oil/biomass bodies took place but due to the presence of the membrane biomass washout did not occur. The mixed liquor was pseudo plastic and the apparent viscosity and flow behavior index generally increased with salt concentration. The use of a MBR system containing a moderately halophilic bacterial consortium resulted in fairly good organic and turbidity removal from synthetic PWs containing 100–250 g/L NaCl with no associated membrane fouling. Organic accumulation in the mixed liquor was observed at all salt concentrations but main removal mechanism was always biodegradation. At the lower end of the salt concentration SVI rise was observed whereas at the higher end oil/biomass bodies formed; in both cases the presence of microfiltration membrane prevented biomass washout. Apparent viscosity and flow behavior index of mixed liquor increased with salt concentration.

Capodic et al. [22] studied pilot scale experiment with MBR operated in intermittent aeration condition: Analysis of biological performance. The effect of intermittent aeration (IA) on a MBR system was investigated. The study was aimed at analyzing different working conditions and the influence of different IA cycles on the biological performance of the MBR pilot plant, in terms of organic carbon and ammonium removal as well as extracellular polymeric substances (EPSs) production. The membrane modules were placed in a separate compartment, continuously aerated. This configuration allowed to disconnect from the filtration stage the biological phenomena occurring into the IA bioreactor. The observed results highlighted good efficiencies, in terms of organic carbon

and ammonium removal. It was noticed a significant soluble microbial products (SMPs) release, likely related to the higher metabolic stress that anoxic conditions exerted on the biomass. However, the proposed configuration, with the membranes in a separate compartment, allowed to reduce the EPSs in the membrane tank even during the non-aerated phase, thus lowering fouling development. The MBR pilot showed high performances in terms of COD and BOD5 removals, irrespective of cycle characteristics. Significant release of SMP occurred during the anoxic phase, likely due to a stress condition on the biomass. Therefore, extended durations of non-aerated phases might enhance denitrification but could have a significant impact on membrane fouling related to EPS production.

Hu et al. [23] studied effects of alkalinity on membrane bioreactors for reject water treatment: Performance improvement, fouling mitigation and microbial structures. Two submerged membrane bioreactors (MBRs) for reject water treatment were operated to investigate effects of sodium bicarbonate (SB) addition on enhancing process performance and mitigating membrane fouling. Results showed that SB addition enhanced average removal efficiencies of COD and NH4-N by 14.6% and 38.3%, respectively. With SB addition, the extracellular polymeric substances (EPS) content in activated sludge increased, but those in membrane foulants greatly decreased. Gel permeation chromatography analysis demonstrated that EPS in MBRs for reject water treatment had much larger molecular weight (MW) and broader MW distribution than those in MBRs for municipal wastewater treatment. The fouling mitigation by SB was attributed to a deprotonation mechanism reduced EPS adsorption on negatively charged membrane surfaces, and improvement of degradation efficiency of macromolecular organic matters..

SB addition into the MBR for reject water treatment enhanced average removal efficiencies of COD and NH4-N by 14.6% and 38.3%, respectively. With SB addition, contents of EPS increased in AS, but greatly decreased in MF. The fouling mitigation by SB was attributed to a deprotonation mechanism reduced EPS adsorption on negatively charged membrane surfaces. GPC analysis demonstrated that EPS in MBRs for reject water treatment had much larger MW and broader MW distribution than those for municipal wastewater. SB addition also increased microbial abundance, enriched nitrifying bacteria, and converted predominant AOB genus from Nitrosomonas to Nitrosospira.

2.5 Hybrid Systems

Selecting the best method is based on many factors such as the characteristics of the produced water, the space availability and the reuse and discharge plans. Eventually, a unique method or treatment system cannot be used with any produced water from any oilfield around the world. Every year, the governments and the environmental agencies implement new and strict standards for discharging treated produced water to the environment. Newly developed hybrid systems can help to improve the final treatment effluent to meet the strict requirements. The objective of processes combination is to overcome the operational problems and limitations that each processes present when used separately.

Murray-Gulde et al. [24] studied performance of a hybrid reverse osmosis-constructed wetland treatment system for brackish oil field produced water. Using constructed wetlands, produced waters from oil fields (i.e., waters that have been in contact with oil in situ) can be treated to enhance water quality for irrigation purposes, or subsequent

discharge to receiving aquatic systems. In produced water containing elevated levels of salt (i.e., brackish-produced waters), the ability to decrease the conductivity of the produced water may influence potential reuse of the water for irrigation purposes. Treatment of produced water from oil production wells using a hybrid reverse osmosis-constructed wetland treatment system met the overall objective of this study, which was to achieve water suitable for irrigation or discharge to surface waters, except additional treatment will be necessary to control boron levels if the water is to be used for irrigation. The pilot-scale RO unit effectively decreased conductivity and TDS in this brackish-produced water (98% and 96% removal, respectively) and reduced the concentration of several water quality parameters between 83% and 100%.

Da Silva et al. [25] studied oil removal from produced water by conjugation of flotation and photo-Fenton processes. The experiments were conducted in a column flotation and annular lamp reactor for induced air flotation and photo-degradation steps, respectively. A nonionic surfactant was used as a flotation agent. The flotation experimental data were analyzed in terms of a first-order kinetic rate model. Two experimental designs were employed to evaluate the oil removal efficiency: fractional experimental design and central composite rotational design (CCRD). Overall oil removal of 99% was reached in the optimum experimental condition after 10 min of flotation followed by 45 min of photo-Fenton. The results of the conjugation of induced air flotation and photo-Fenton processes allowed meeting the wastewater limits established by the legislations for disposal. The experimental results of oil removal by flotation were described by a first-order kinetic model. For the evaluated surfactant concentrations, the highest removal rate (k ¼ 0.772 min^{-1}) was obtained at $4.06.10^{-3}$ mM and represents 86% of TOG reduction after 4 min of

flotation. With respect to the photo-Fenton step, the highest oil removal achieved was 84% after 45 min of reaction, using 0.44 mM and 10 mM of ferrous ions and hydrogen peroxide, respectively. The best condition found for the integrated processes was 10 min of flotation followed by 45 min of photo-Fenton with overall TOG reduction of 99%, which resulted in only 5 ppm of TOG in the treated effluent. The integration of the flotation and photo-Fenton processes proved to be very effective in reducing TOG in oilfield produced water.

Motta et al. [26] studied produced water treatment for oil removal by an integration of coalescer bed and microfiltration membrane processes. The coalescer bed was formed by cationic exchange resins, working in up flow condition. The MF unit used a polyetherimide hollow fiber submerged module with a permeation area of 0.5 m^2. The investigations were conducted by using synthetic OPW with oil concentrations of 200–400 mg L^{-1} and oil droplet diameters from 3 to 8 μm. In the coalescer bed the fluid velocity ranged from 4.4 to 17.4 m/h and the bed height was 5 cm. The water recovery rate in MF was kept in the range of 0.75–0.90 by using a transmembrane pressure varying from 12 to 30 kPa. The coalecer bed worked in steady state conditions, reaching an efficiency of 35–52%. The overall efficiency of the integrated process reached 93–100% and the oil contents ranged from 0.1 to 14.8 mg L^{-1}. These results indicate that the effluent in the integrated process has quality not only for injection, but also for reuse purposes. The results showed that the integrated process comprising a coalescer bed and microfiltration membranes may represent an important alternative for oil removal from OPW. Regarding the final effluent quality, an important result observed was that both processes contribute to the removal of oil from the OPW sample. For the conditions and parameters tested, the coalescer bed

contributed to remove up to half of the oil content from the OPW, while the membranes contributed to removing almost all of the remaining oil. The total system efficiency varied between 93.0% and 100%, which corresponded to an O&G concentration in the final effluent between 0.1 and 14.8 mg L^{-1}. These values represent excellent results for both purposes, disposal into the sea or injection. In fact, the quality of the treated OPW is good to the point that it is constituted in an indication of the possibility of its reuse in productive activities surface. Regarding the stability of the integrated process, it was observed that, for its first part (the coalescer bed), the Ec varied according to three distinct stages, the first one being strongly ascending, the second mildly descendent and the third constant over time.

Guo and Li [27] studied treatment of oilfield produced water by anaerobic process coupled with micro-electrolysis. Treatment of oilfield produced water was investigated using an anaerobic process coupled with micro-electrolysis (ME), focusing on changes in COD and biodegradability. Results showed that COD exhibited an abnormal change in the single anaerobic system in which it increased within the first 168 hr, but then decreased to 222 mg/L after 360 hr. The biological oxygen demand (five-day) (BOD_5)/COD ratio of the water increased from 0.05 to 0.15. Hydrocarbons in the wastewater, such as pectin, degraded to small molecules during the hydrolytic acidification process. Comparatively, the effect of ME was also investigated. The COD underwent a slight decrease and the BOD5/COD ratio of the water improved from 0.05 to 0.17 after ME. Removal of COD was 38.3% under the idealized ME conditions (pH 6.0), using iron and active carbon (80 and 40 g/L, respectively). Coupling the anaerobic process with ME accelerated the COD removal ratio (average removal was 53.3%). Gas chromatography/mass spectrometry was

used to analyze organic species conversion. In the single anaerobic system, the COD and BOD5 both increased initially over time and then decreased gradually. The BOD5/COD ratio of the produced water increased upon treatment. The organic composition changed during anaerobic processing. Coupling anaerobic treatment with ME accelerated the conversion and biodegradation processes. GC/MS analysis revealed that combined treatment was particularly effective for the conversion of large-molecule organics to smaller ones that were biodegraded only partially. The combined process was very effective at transforming most of the organic pollutants found in the heavy oilfield produced water.

Younker and Walsh [28] studied bench-scale investigation of an integrated adsorption–coagulation–dissolved air flotation process for produced water treatment. Investigation was done at bench scale, chemical coagulation with ferric chloride (FeCl3) and adsorption with organoclay (OC) in a completely stirred tank reactor (CSTR) configuration as pre-treatment for dissolved air flotation (DAF) for the removal of dissolved and dispersed oils from produced water. The integrated process was evaluated and compared to the individual processes of coagulation-DAF, adsorption-DAF and DAF without pre-treatment for the removal of dispersed oil, naphthalene and phenol from synthetic produced water. Concentrations of dispersed oil in clarified water were reduced, from an initial concentration of 100 mg/L, to concentrations as low as 10±1.6 mg/L after coagulation with $FeCl_3$ ($FeCl_3$-DAF), 15 ±1.2 mg/L after adsorption with OC (OC-DAF), and 7 ± 1.4 mg/L after the integrated process (OC$FeCl_3$- DAF). From an initial naphthalene concentration of 1 mg/L, both the adsorption (OC-DAF) and integrated process (OC-$FeCl_3$) achieved

clarified naphthalene concentrations of 0.11±0.01 mg/L, representing a significant improvement over the 0.53±0.03 mg/L achieved by coagulation treatment ($FeCl_3$-DAF).

Nguyen et al. [29] studied removal of trace organic contaminants by a membrane bioreactor–granular activated carbon (MBR–GAC) system. A synthetic wastewater was used to ensure a consistent influent composition. The MBR showed stable and good performance with respect to all key basic water quality and operating parameter. Turbidity of MBR permeate was consistently below 0.2 NTU during the entire period of operation. In addition, a stable TOC removal (97–99%) was achieved and TOC concentration of the permeate was typically less than 5 mg/L. The MBR system was operated under aerobic conditions and, therefore, is not expected to have high nitrogen removal via denitrification. Little or no removal was observed for carbamazepine, diclofenac and fenoprop, while 80–99% removal of all five steroid hormones and four alkyl phenolic trace organics could be observed. Results confirm that MBR treatment can effectively remove hydrophobic (logD > 3.2) and readily biodegradable trace organics but is less effective for the removal of hydrophilic and persistent compounds. GAC post-treatment was observed to significantly complement MBR treatment to obtain high overall removal of less hydrophobic and biologically persistent trace organics. However, breakthrough of diclofenac, whose concentration in the GAC effluent was monitored for extended period, indicated that strict monitoring should be applied over the lifetime of the GAC column to detect the breakthrough point of hydrophilic and persistent compounds which have low removal by MBR treatment.

2.6 Concluding Remarks

From the literature review, the following conclusions can be drawn:

- Produced water is conventionally treated through different physical, chemical, and biological methods.
- Major research efforts in the future could focus on the optimization of current technologies and use of combined physico-chemical and/or biological treatment of produced water in order to comply with reuse and discharge limits.
- Both Electrocoagulation and membrane bioreactors are widely used for the treatment of produced water and are showing a relatively high treatment efficiencies.
- Nowadays, it is possible to find a large amount of research work about both electrocoagulation and MBR processes in the literature, not only in experimental terms but also in theoretical ones. However, there is no information so far about these two processes operating in an integrated hybrid system.

CHAPTER 3

OBJECTIVES

Based on the literature review and up to the knowledge of the investigator, there is a lack of information on a hybrid system that consists of electrocoagulation unit and an immersed membrane bioreactor unit. The main objective of the research is to test the effectiveness of the electrocoagulation-membrane bioreactor hybrid system in treating oilfield produced water to the worldwide standards. The specific objectives are to:

(1) Investigate the effect of two different electrode materials (steel and aluminum) on the removal efficiency performance of the electrochemical cell. (Optimization of the electrochemical cell)

(2) Investigate the effect of current density on the removal efficiency performance of the electrochemical cell. (Optimization of the electrochemical cell)

(3) Investigate the effect of detention time on the removal efficiency performance of the electrochemical cell. (Optimization of the electrochemical cell)

(4) Investigate the fouling behavior of the membrane process. (Optimization of the flux)

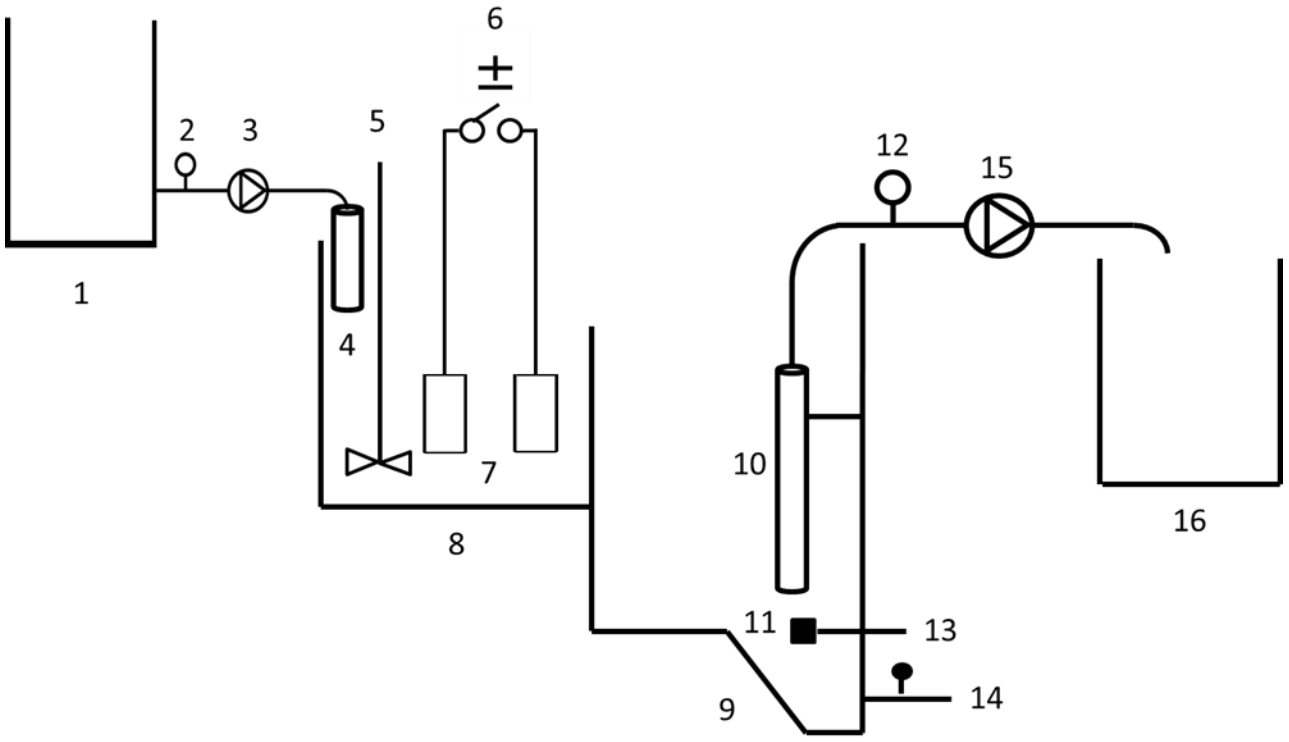
(5) Investigate the effect of oil content in the synthetic produced water on the performance of the electrocoagulation-membrane bioreactor hybrid system. (Optimization of the hybrid system)

CHAPTER 4

METHODOLOGY

4.1 Experimental Setup

The experimental setup comprises feed tank, electrochemical cell tank, immersed membrane tank, membrane module and permeate tank. The tanks are made of Plexiglas sheets. Figure 1 shows a schematic diagram of the experimental setup.



1. Feed Tank
2. Pressure Gauge
3. Pump
4. Level Control Device
5. Mixer
6. DC Power Supply
7. Electrodes
8. Electrochemical Cell
9. Immersed Membrane Tank
10. Membrane Module
11. Air Diffuser
12. Pressure Gauge
13. Air Supply
14. Waste Line
15. Pump
16. Permeate Tank

Figure 4.1 Schematic Diagram of the Experimental Setup

4.2 Membrane Specifications

A lab scale submerged membrane module is used. The membrane has nominal membrane surface area of 0.047 m^2. The membrane can be operated with a maximum trans membrane pressure of 62 kPa (9.0 psig). The membrane can be operated with a pump capacity from 0 to 150 milliliters/minute. A summary of the main membrane specifications are in Table XX and the full membrane specifications can be found in the installation and operating manual in Appendix B.

Table 4.1 Membrane Characteristics

Parameter	Value
Configuration	Hollow Fiber
Material	PVDF
Pore Size	0.035 µm
Nominal Permeate Flow	20 $l/m^2.hr$
Maximum TM Backwash Pressure	8.0 psi
Typical Operating TMP	10-50 kPa (1.0-7.0 psi)
Maximum Operating Temperature	40°C (104°F)
Filtration Direction	Outside to Inside
Membrane Length	175 mm
Nominal Surface Area	0.047 m^2
Maximum Aeration Flow per Module	1.8 m^3/h (1 scfm)

4.3 Electrodes

Both the Aluminum and the Stainless Steel electrodes used in the experiments were manufactured in a workshop in Al-Khudhria Industrial Area, Dammam. The Aluminum electrodes were attached to the holders using a compression screws whereas welding was used to attach the Stainless Steel electrodes to the holders. The electrodes are 10 x 5 cm2.

Figure 4.2 Stainless Steel Electrode (left) Aluminum Electrode (right)

4.4 Synthetic Wastewater

Under the supervision of the Civil and Environmental Engineering Department and the Office of the Supervisor of General Services, Saudi Aramco, a Saudi Arabian national petroleum and natural gas company based in Dhahran, were contacted to deliver a produced water samples from Abqiq Gas and Oil Separation Plant, Abqiq GOSP. The produced water characterized and a synthetic water with similar characteristics will be used. Table 4.2 summaries the synthetic wastewater characteristics. Sea water collected from Azizia Peach, Khobar, Saudi Arabia were used to synthetize the produced water used in all the experiment. A rapid mixer was used for mixing the oil with water and each Liter of sea water was mixed for 4 minutes.

Table 4.2 Synthetic Wastewater Characteristics

Parameter	Value
pH	7.20
Conductivity	50 mS/cm
Salinity	55 g/L
Oil & Grease	100, 150 and 200 mg/L

4.5 Design of Experiment

The experiment will consist of four main stages:

i. Synthetizing the waste (see section 4.3).

ii. Electrocoagulation stage using an electrochemical setup will be used to find the optimum condition for the treatment of the produced water. The electrochemical setup will consist of an electrochemical cell, a DC power supply, a pair of electrodes dipped in the wastewater in a glass beaker. In this stage, three current densities (5, 10 and 30 mA/cm^2) and six different contact times (5, 15, 30, 60, 90 and 120 min) will be tested. Moreover, two kinds of electrodes will be used, stainless steel and aluminum. Table 4.3 summarize the experimental design of this stage.

Table 4.3 Design of Electrocoagulation Experiments

Exp. No.	Electrode Type	Current Density (mA/cm^2)	Contact Time (min.)	Exp. No.	Electrode Type	Current Density (mA/cm^2)	Contact Time (min.)
1	Stainless Steel	5	5	19	Aluminum	5	5
2		5	15	20		5	15
3		5	30	21		5	30
4		5	60	22		5	60
5		5	90	23		5	90
6		5	120	24		5	120
7		10	5	25		10	5
8		10	15	26		10	15
9		10	30	27		10	30
10		10	60	28		10	60
11		10	90	29		10	90
12		10	120	30		10	120
13		30	5	31		30	5
14		30	15	32		30	15
15		30	30	33		30	30
16		30	60	34		30	60
17		30	90	35		30	90
18		30	120	36		30	120

iii. Investigate the fouling behavior of the membrane process by running the hybrid system with the following fluxes or until reaching fouling.

Table 4.4 Different Flux Values

No.	Flux (L/hr.m2)	Flow		
		(ml/min)	(L/hr)	(L/day)
1	10.00	6.67	0.40	9.60
2	11.43	7.62	0.46	10.97
3	12.00	8.00	0.48	11.52
4	13.33	8.89	0.53	12.80
5	16.00	10.67	0.64	15.36
6	20.00	13.33	0.80	19.20
7	26.67	17.78	1.07	25.60

iv. Run the hybrid electrocoagulation and membrane bioreactor system with the optimum conditions found from the previous two stages as follow.

Table 4.5 Different Hybrid System Running Stages

Stage	Oil Content	Duration
1	100 mg/L	20 Days
2	150 mg/L	20 Days
3	200 mg/L	20 Days

4.6 Methods of Analysis

The standard methods will be used for the analysis of produced water samples. Table 4.6 shows the different parameters along with the methods or instruments that will be used.

Table 4.6 Methods for Analysis

Parameter	Method
pH	Direct Instrumentation: Digital pH Meter
Conductivity	Direct Instrumentation: Digital Conductivity Meter
MLSS and MLVSS	Standard method: 2005, APHA
COD	A Proposed Method Modification for the Determination of COD in Saline Waters. By (Freire & Sant'Anna, 2010) [42]
TSS	Standard method: 2540 D, APHA
TDS	Standard method: 2540 C, APHA
Turbidity	Turbidity Meter
Oil & Grease	EPA Method 1664, Revision A

CHAPTER 5

RESULTS AND DISCUSSION

5.1 Electrocoagulation Experiments Results

During this stage, 36 individual experiments were performed to study the effect of electrolysis time, current density and electrode material on the removal efficiencies of COD and Turbidity of the synthetic produced water. The effect of current density were studied by varying the range from 5 mA/cm^2 to 30 mA/cm^2. Moreover, six different electrolysis time were investigated with each of the current densities and the electrode materials. Figures 5.1, 5.2, 5.6 and 5.7 show the COD and Turbidity removal efficiencies for the all experiments. Both the COD and Turbidity declined significantly as the current density and the electrolysis time increased. Among these, the reduction of COD using Aluminum electrodes was the highest with a removal efficiency of 65% at 120 minutes and 30 mA/cm^2. With this conditions also, the highest turbidity removal was achieved also with a removal efficiency up to 52%. In general, Aluminum electrodes showed a higher removal efficiencies than Stainless Steel electrodes and the increase in both the electrolysis time and the current density increased the removal efficiency.

According to Faraday's laws of electrolysis, the coagulant generated from the anode was proportional to the total electric charge passing through, that is, the current density. As the current density increased, huge amounts of coagulants were produced to destabilize the colloidal particles, together forming more precipitates which would easily settle down (M.

Tir and N. Moulai-Mostefa, 2008) [43]. On the other hand, higher amount of H_2 would be generated from the cathode due to the higher current. Bubble size decreased with the increasing current density, reported by (Chen, 2004) [44], resulting in the increasing bubble densities and the intense upward flux. Smaller bubbles also provided larger surface area for particle attachment, ensuring high separation efficiency (X. Chen et. al, 2002) [46]. Pollutants were taken away through this flotation process with flocs which were lifted by the large amounts of tiny bubbles. These reasons explained why higher current density created a better reduction for COD and turbidity. Figures 5.3 to 5.5 and 5.8 to 5.10 is showing the relation between the coagulant dosage and the removal efficiency of COD and Turbidity. However, the energy consumption and the operating costs all increased as the current density rose.

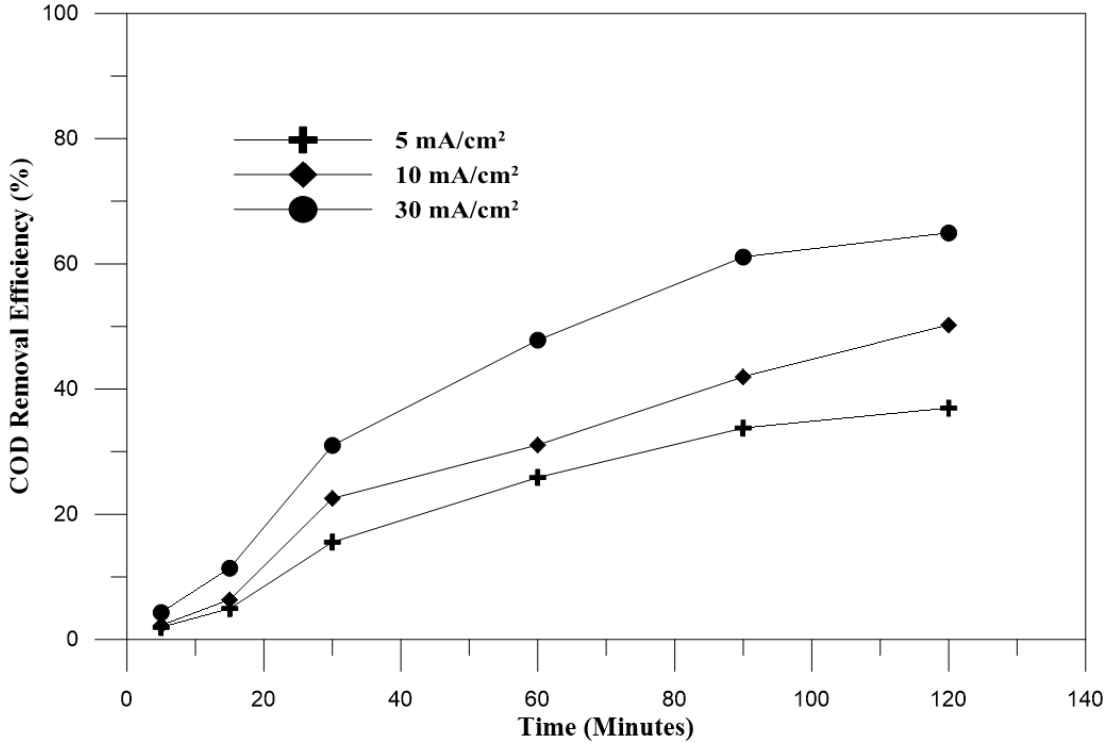


Figure 5.1 COD Removal Efficiency (%) – Aluminum Electrodes – 5, 10 and 30 mA/cm^2

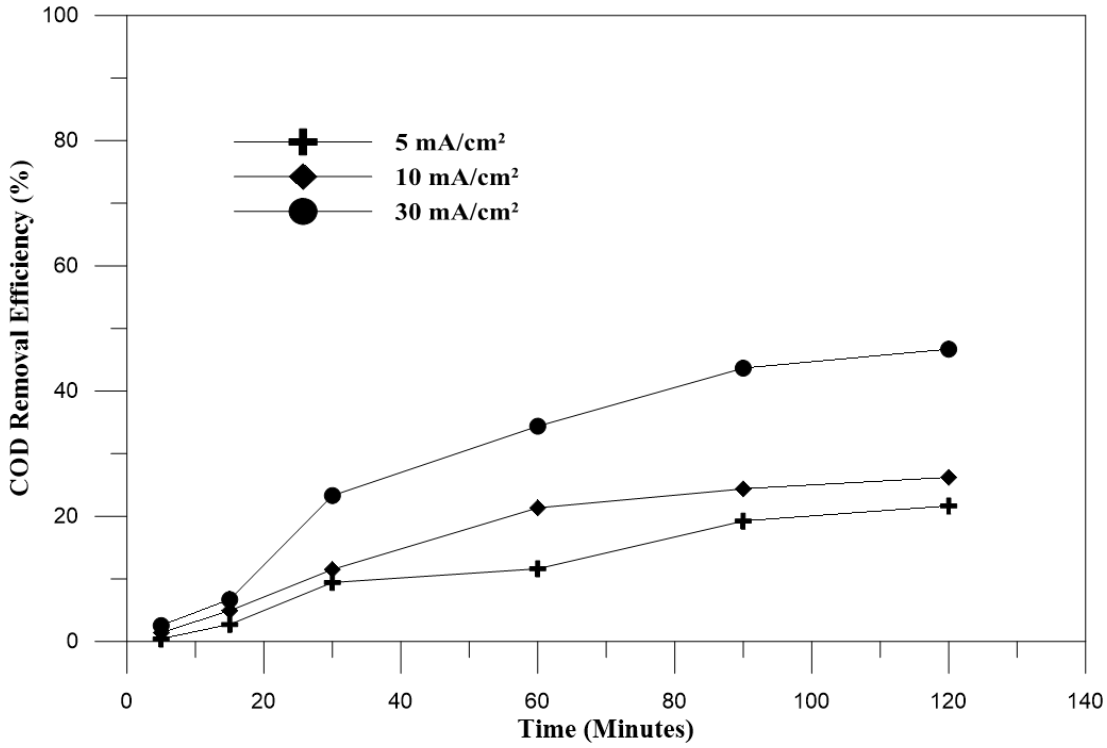


Figure 5.2 COD Removal Efficiency (%) – Stainless Steel Electrodes – 5, 10 and 30 mA/cm^2

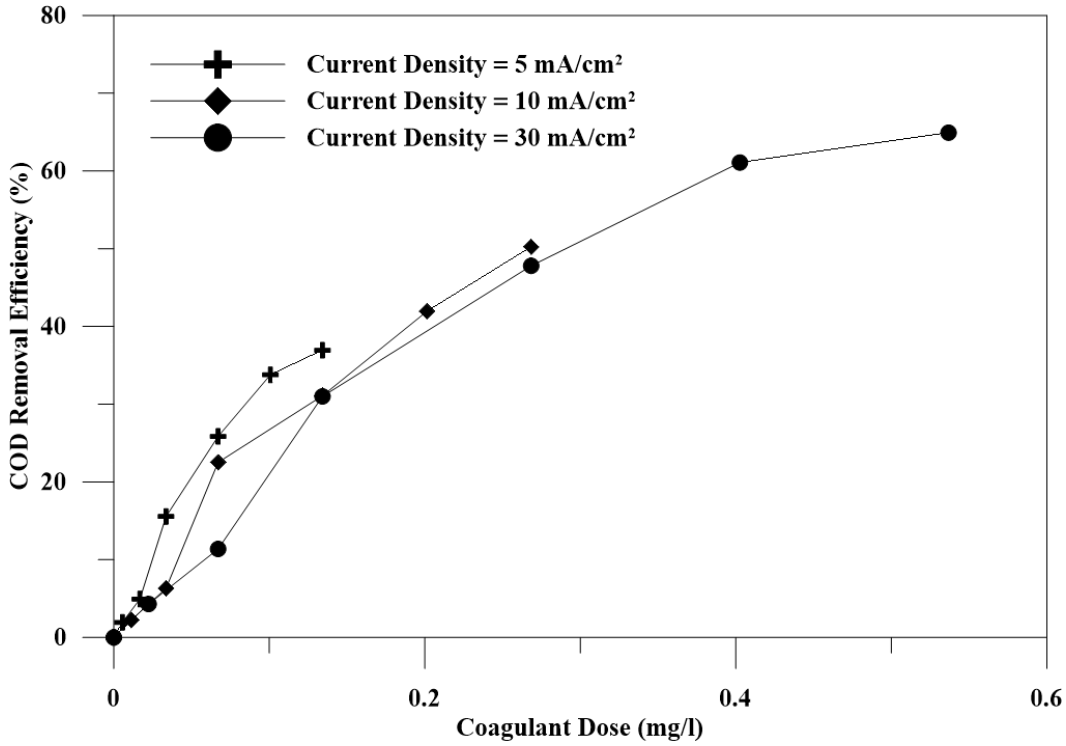


Figure 5.3 COD Removal Efficiency (%) vs Coagulant Dose (mg/L) - Aluminum Electrodes – 5, 10 and 30 mA/cm²

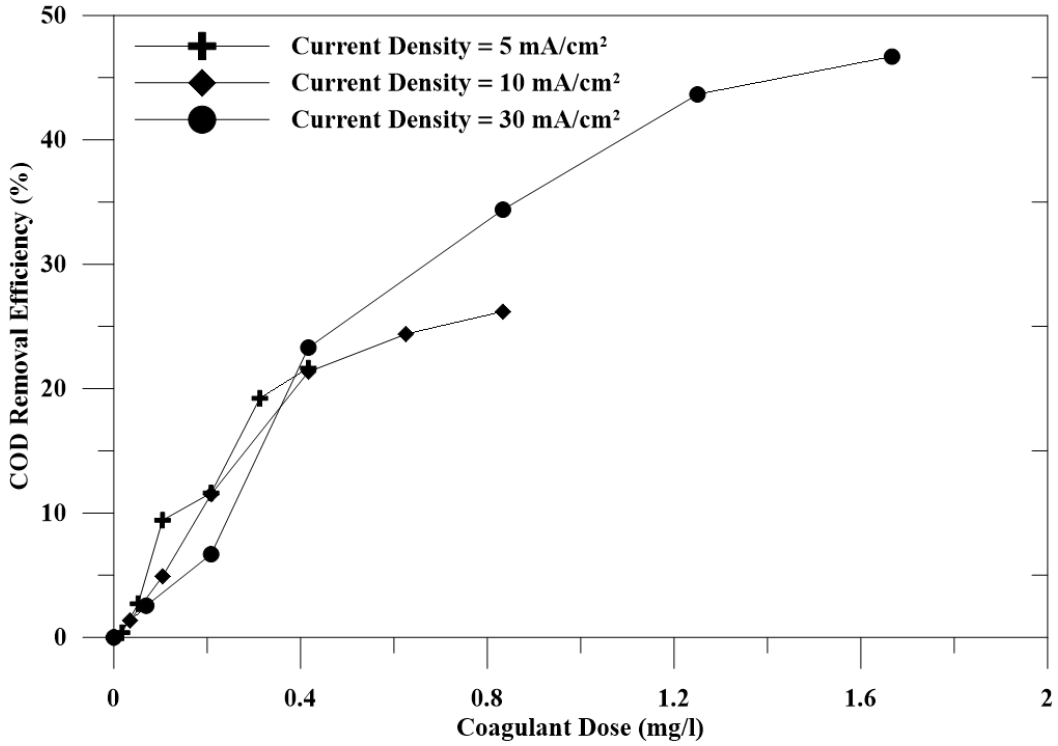


Figure 5.4 COD Removal Efficiency (%) vs Coagulant Dose (mg/L) – Stainless Steel Electrodes – 5, 10 and 30 mA/cm^2

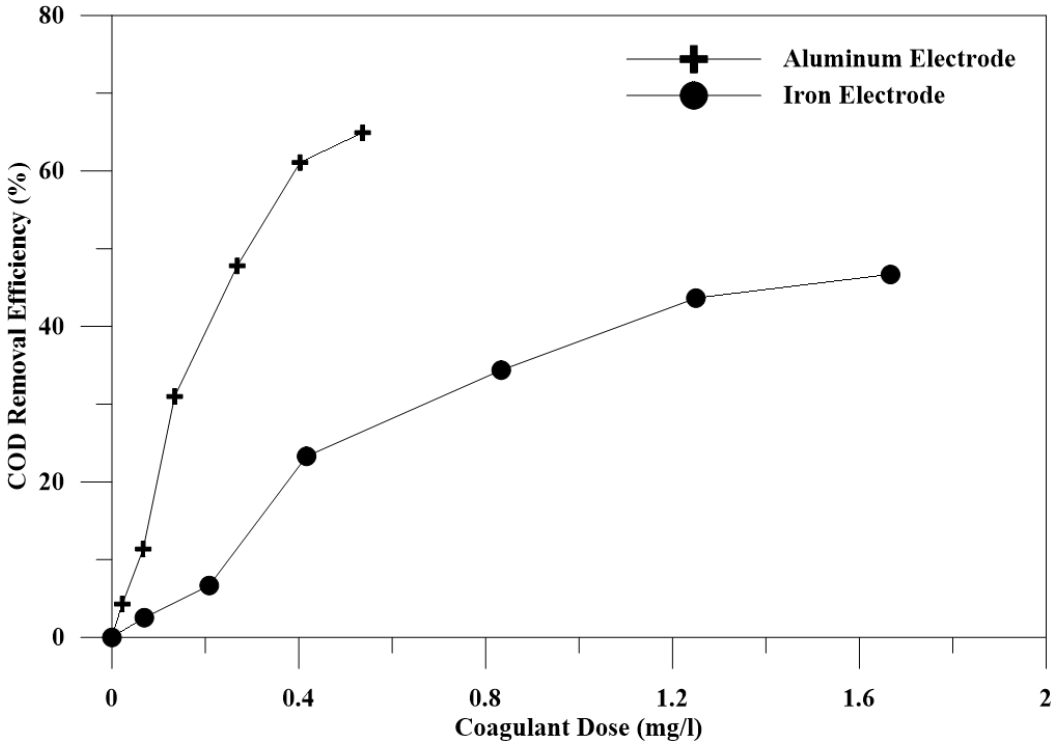


Figure 5.5 COD Removal Efficiency (%) vs Coagulant Dose (mg/L) – Aluminum & Stainless Steel Electrodes – 30 mA/cm^2

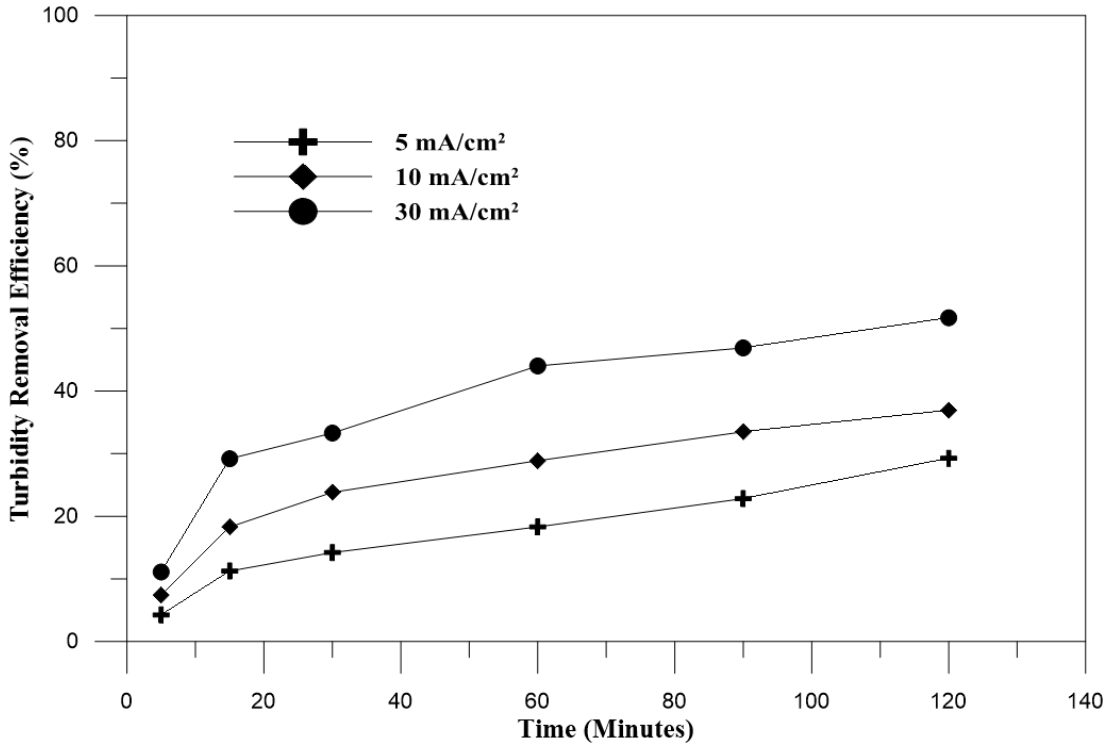


Figure 5.6 Turbidity Removal Efficiency (%) – Aluminum Electrodes – 5, 10 and 30 mA/cm^2

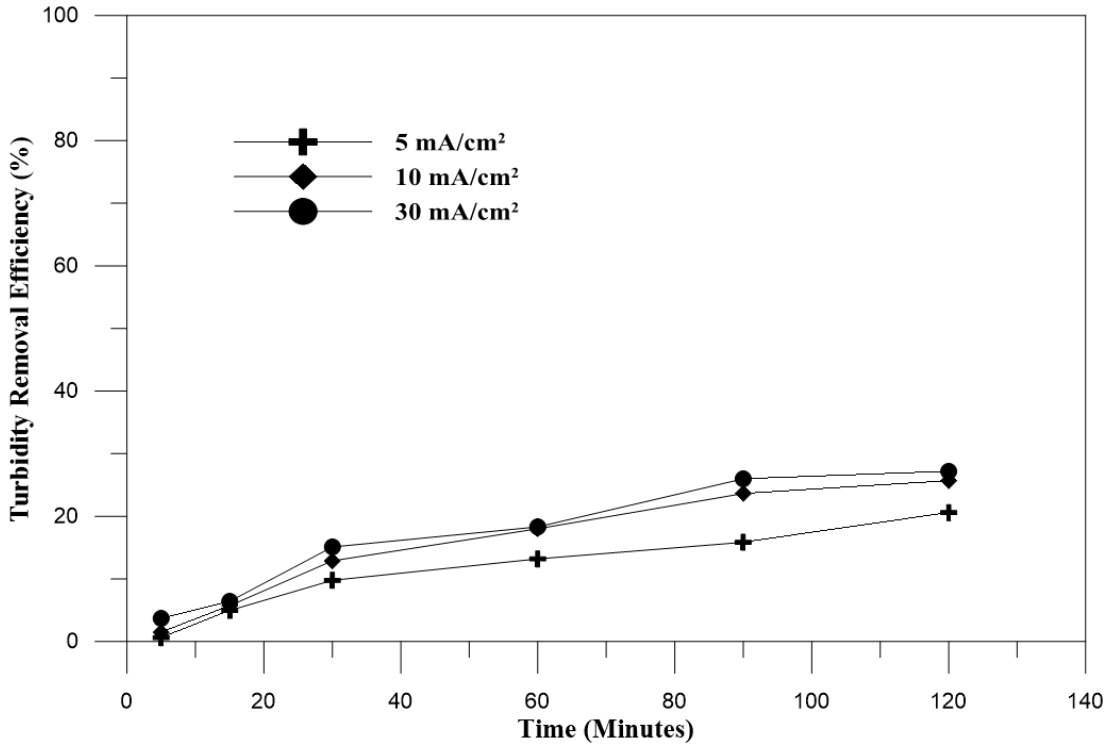


Figure 5.7 Turbidity Removal Efficiency (%) – Stainless Steel Electrodes – 5, 10 and 30 mA/cm^2

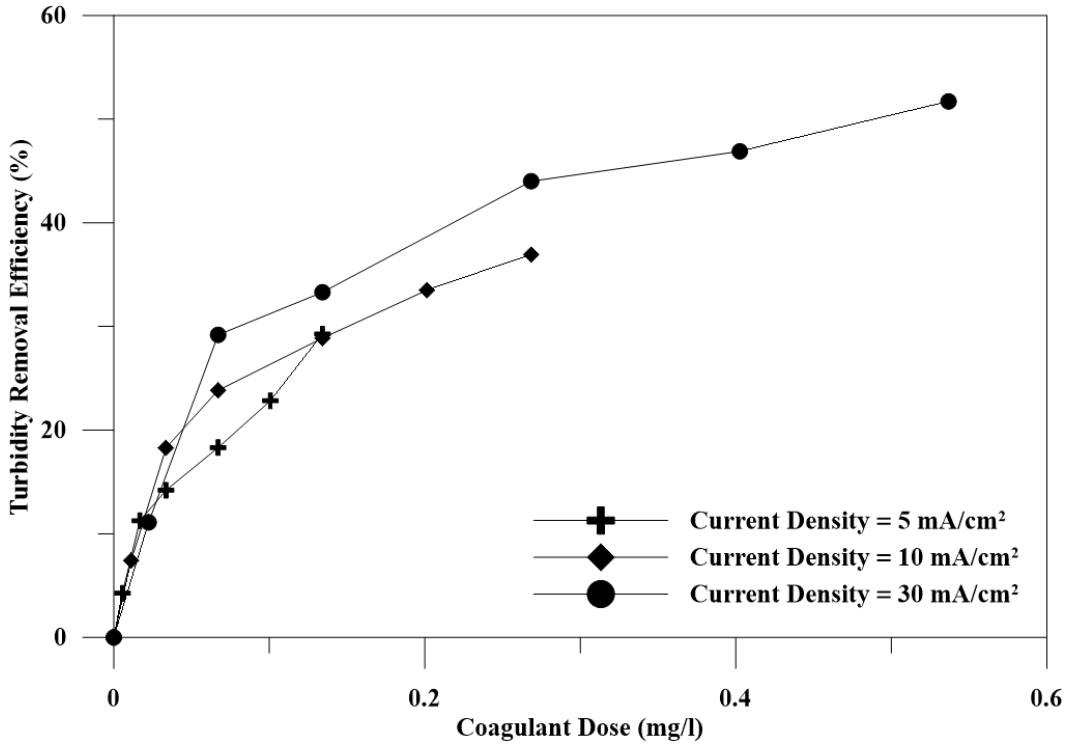


Figure 5.8 Turbidity Removal Efficiency (%) vs Coagulant Dose (mg/L) – Aluminum Electrodes – 5, 10 and 30 mA/cm^2

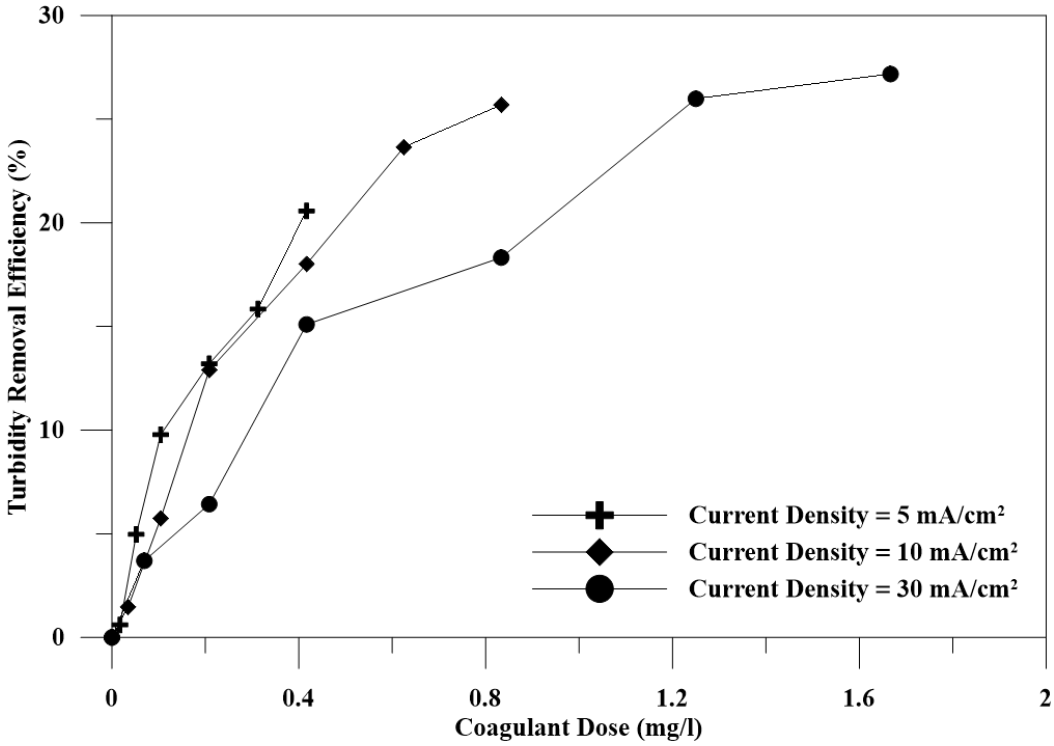


Figure 5.9 Turbidity Removal Efficiency (%) vs Coagulant Dose (mg/L) – Stainless Steel Electrodes – 5, 10 and 30 mA/cm^2

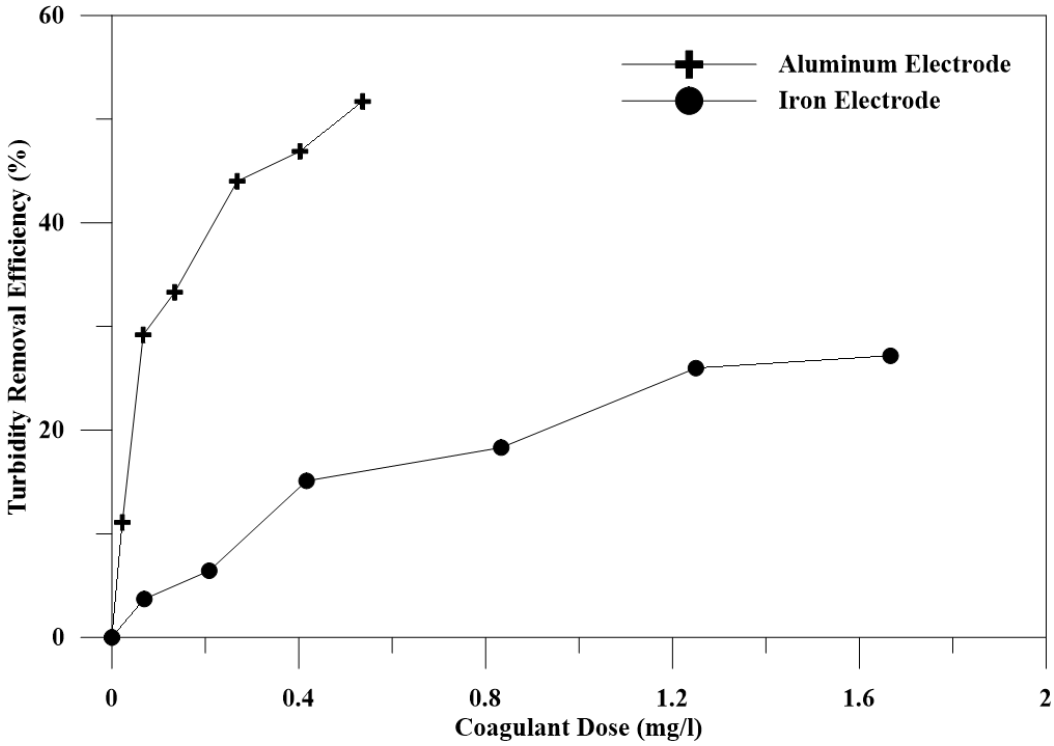


Figure 5.10 Turbidity Removal Efficiency (%) vs Coagulant Dose (mg/L) – Aluminum & Stainless Steel Electrodes – 30 mA/cm^2

5.2 Membrane Fouling Performance

In this stage the hybrid system was operated with five different fluxes (10, 11.43, 13.33, 16 and 20 L/m^2.hr) each for four days to study the performance of the membrane (see section 4.2 and Appendix B for detailed membrane specifications). At the beginning of each stage the MLVSS was adjusted to 5000 mg/L. The pressure was monitored closely at the beginning of each stage (at 0.5, 1, 2, 3 and 6 hours) and every 12 hours for the rest of each experiment.

For the first four experiments (flux = 10, 11.43, 13.33 and 16 L/m^2.hr) the pressure values increased with time until a steady state reached after 48 hours of operation as showed in Figures 5.13. At the fifth experiment a membrane fouling occurred (7.00 psi) after 24 hours of operation. A backwash for the membrane is done for 10 minutes and the pressure was monitored closely. A second membrane fouling occurred at 48 hours and the same procedure was repeated. The next remaining days of the operation showed that a membrane fouling occurred every 24 hours (Figure 5.13). The increase in the pressure and the drop in the flux value could be attributed to either a decrease in the membrane area due to clogging of membrane pores or an increase in the hydraulic resistance to filtration. This increase in hydraulic resistance can be caused either by narrowing of the pores of the membrane or by cake formation on the membrane surface. The total hydraulic resistance comprises resistance caused by internal membrane fouling, and resistance caused by deposition of particles and or colloids on the primary membrane surface. Other reasons which could have caused membrane clogging include the size of the pores of the membrane, the surface

charge of the membrane, the mechanism of adsorption of particles onto the membrane surface and the hydrophobic or hydrophilic nature of the membrane surface.

Almost an identical result to the pressure results is shown in Figure 5.12 for the membrane resistance for the five experiments. The hydraulic resistance is caused by deposition of oil and particles with time. At the last 48 hrs of the four experiments (Flux of 11.43, 13.33, 16 and 20 $L/m^2.hr$) a hydraulic resistance value of 0.7×10^{-10} per meter were reached. The increase in the hydraulic resistance can be attributed to fouling of the membrane. It is worth to mention that membrane performance is dependent on more complex interactions between particle size, membrane surface, and performance history, as well as a number of other factors.

The collected permeate cumulative volume with respect to running time is shown in Figure 5.11 for the five experiments. The figure clearly demonstrates an increase in the cumulative volume with the increase in the running time for all the five fluxes tested.

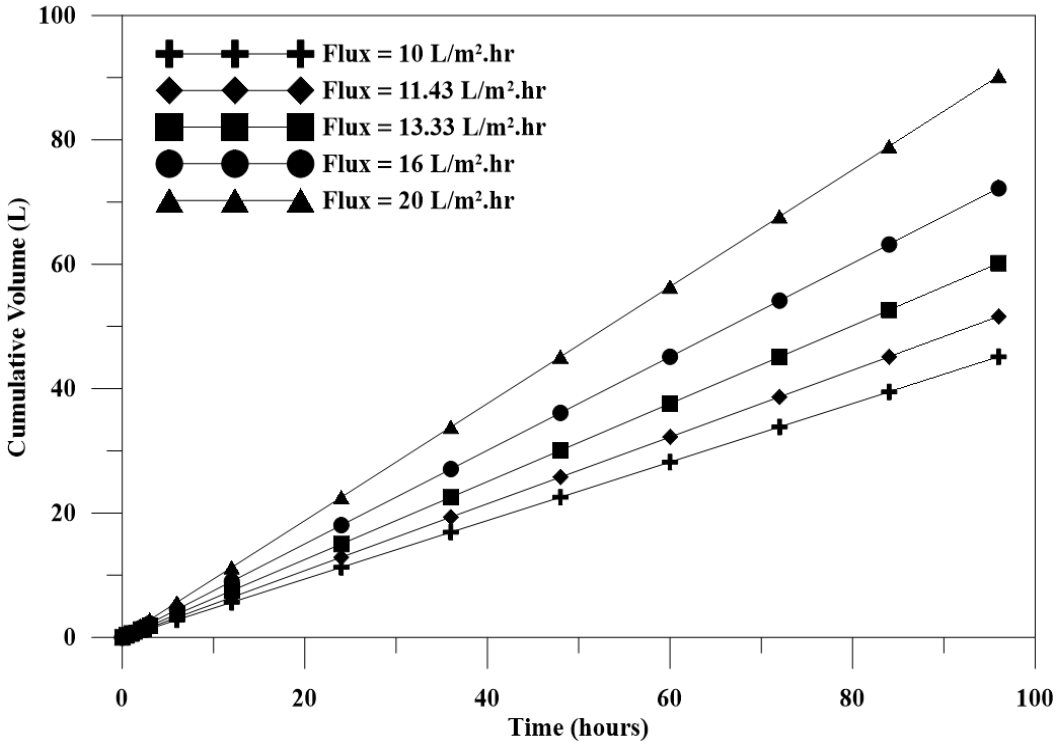


Figure 5.11 Cumulative Volume (L) vs Time (hours) – Membrane Fouling Performance Studies

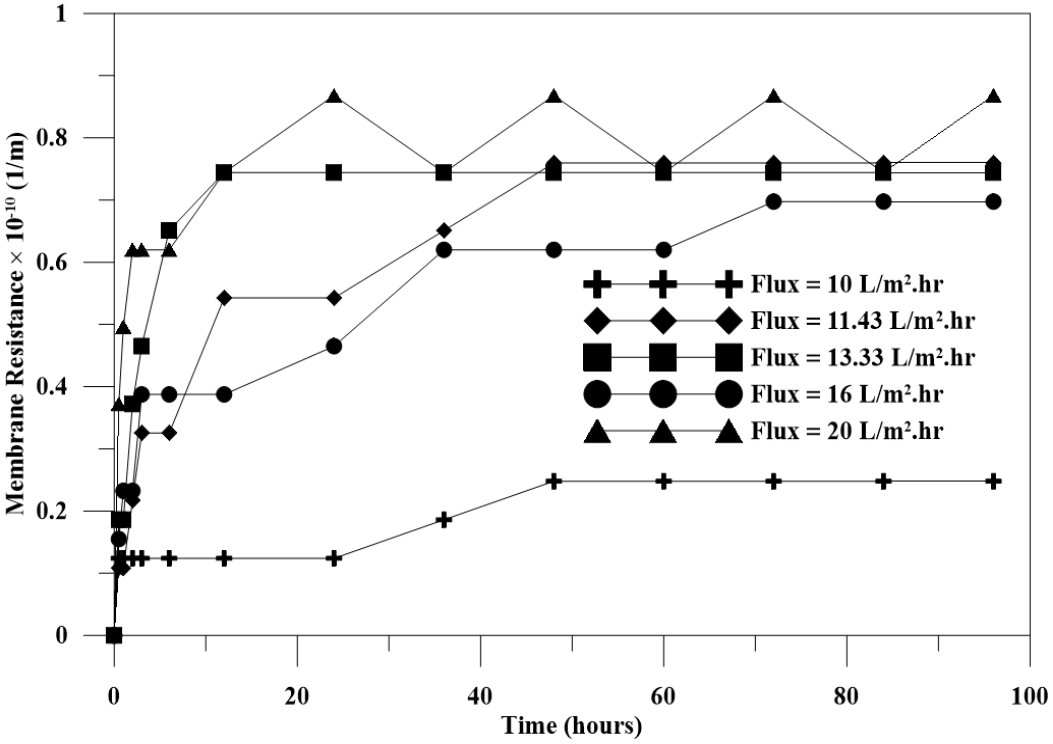


Figure 5.12 Membrane Resistance x 10^{-10} (L/m) vs Time (hours) – Membrane Fouling Performance Studies

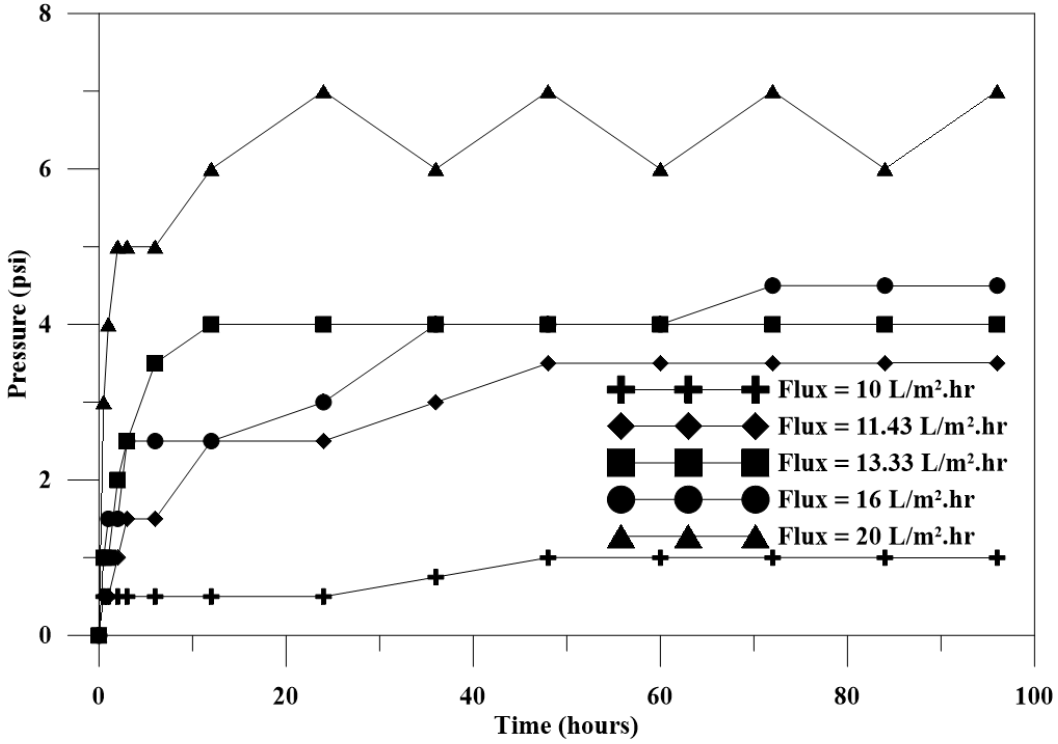


Figure 5.13 Pressure (psi) vs Time (hours) – Membrane Fouling Performance Studies

5.3 Hybrid System Performance

After the electrocoagulation experiments and the membrane fouling experiments. A flux of 12 L/hr.m^2 and a current density of 30 mA/cm^2 were selected to run the three stages of the hybrid system.

5.3.1 Acclimatization

Microorganisms were acclimatized to the nutrients and to the high oil concentrations. The nutrients used for the acclimatization process were Nutrient Broth in addition to crude oil from a petroleum refinery. At the initial stage of acclimatization, higher concentration of nutrient and lower concentration of crude oil were used. With the progression of time, the oil concentration was increased to reduce the dependency on the nutrient which was not fully eliminated. The biomass was acclimatized to the nutrient and oil for 90 days.

5.3.2 Membrane Fouling Performance

During the 60 days of operation of the hybrid system the actual flux was monitored and calculated twice a day. Figure 5.14 show the actual flux for the three stages. It is clear that some decreases in the actual flux took place during different times of the operation. The actual flux value was adjusted by gradually increasing the speed of the peristaltic pump operating in the hybrid system to reach the desired value of flux at 12L/hr.m^2. The peristaltic pump then left to operate at the same speed until another decrease in the actual flux occurred. The flux stability could be attributed to presence of air diffusers at the bottom of the membrane module. The air diffusers were provided to prevent or even reduce fouling

of the membrane and serve the purpose of mixing the reactor contents and maintain aerobic conditions in the reactor. The other reason could be the low TMP that was maintained throughout the experimental period.

The pressure was also monitored during the operation of the hybrid system to make sure that the system is operating below the maximum operating pressure of the membrane (6.0 psi). Figure 5.15 show a 12 hours based data of the psi during the run of the three stages. The pressure reached a constant value of 3.5 to 4.0 psi from the second day of operation to the end of each stages and no backwashing was needed during the 60 days run of the hybrid system.

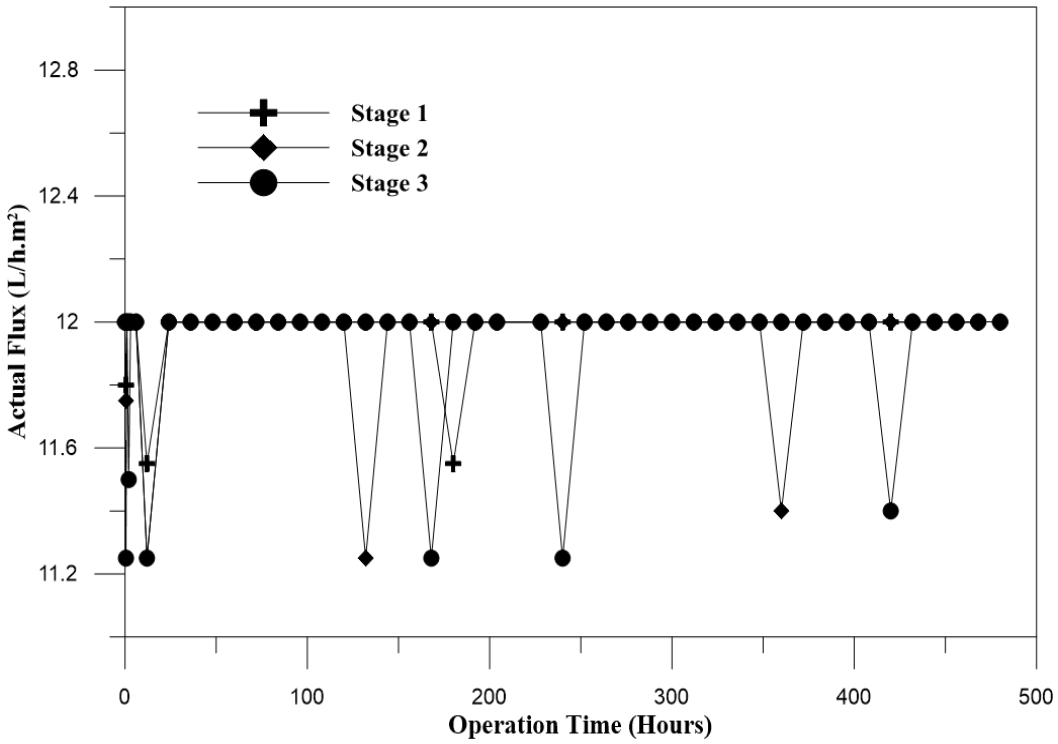


Figure 5.14 Actual Flux (L/h.m^2) vs Operation Time (hours) – Membrane Fouling Performance - Hybrid System

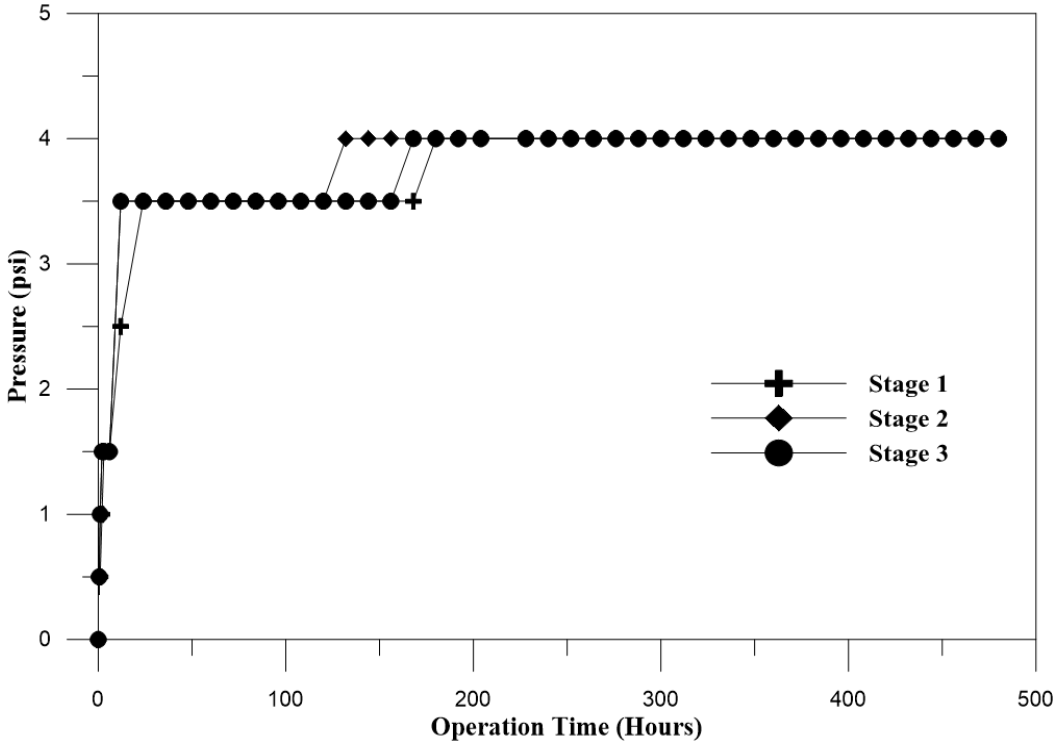


Figure 5.15 Pressure (psi) vs Operation Time (hours) – Membrane Fouling Performance - Hybrid System

5.3.3 MLSS & MLVSS and Bacterial Colony Plate Count

At the beginning of each of the three stages of the hybrid system, the MLVSS adjusted to 5000 mg/L. During the experimental study, no sludge was withdrawn from the bioreactor. The change in MLSS, MLVSS and bacterial counts during operation of the hybrid system is presented in Figures from 5.16 to 5.20. One thing to note from the results is the inconsistency between the trend of change of bacterial counts and that of MLSS and MLVSS with increasing the oil content from 100 to 200 mg/L during the three stages of the hybrid system. The increasing trend of change of bacterial count with the oil content is in line with previous studies with activated sludge and other wastewaters in both MBR (Huang et al., 2011) [46] and MSBR (Pajoum Shariati et al., 2011) [47].

In MBR studies, MLSS and MLVSS are usually used as indicators of biomass concentration. However, according to a previous study by (Abdollahzadeh Sharghi and Bonakdarpour, 2013) [48] when inorganic matter and O&G accumulate inside the MBR, MLSS and MLVSS are no longer exact indicators of biomass concentration. Therefore, bacterial colony count maybe used as a more direct measure of the halophilic bacterial population. Although they do not show up the concentration of any nonculturable bacterial species present in the bacterial consortium, the bacterial counts are the most direct measure of the bacterial population in the MBR. The fact that during the run in the present study trend of change of MLSS did not follow that of bacterial counts can be explained by the pronounced change in the inorganic content of MLSS as evidenced by the rise in the value of MLVSS/MLSS ratio from a value of 78% in the beginning of stage 1 to a value of 85% at the end of stage 3 (Figure 5.19).

The variation in MLSS and MLVSS during the three stages are presented in figures 5.16, 5.17 and 5.18. The results reveal that the trend of change of MLSS and MLVSS with the time of operation of the membrane bioreactor during the second and the third stages does not follow that of bacterial colony plate count. This discrepancy can be explained by the fact that MLSS reflects both the inorganic and organic content of the mixed liquor, whereas MLVSS concentration reflect the oil adsorbed onto the bacterial flocs as well as the bacterial biomass concentration.

On the other hand, the trend of change of MLVSS data is influenced both by the biomass concentration as well as the mixed liquor O&G, which showed great variation with time (Figures 5.16 to 5.18). Therefore, under the conditions employed in the MBR run, the trend of change of MLSS and MLVSS were not exact representatives of the concentration of biomass inside the MBR. For this reason, bacterial colony count data were presented.

A sample from the MBR was taken on day 1, 5, 10, 15 and 20 of operation of the hybrid system in the three stages and the average of six bacterial colony plates count is taking into account. In stage 1, the average plate count increased from 3370 CFU/ml at day 1 to 4430 CFU/ml at day 20 (Figure 5.20). In stages 2 and 3, the increasing trend were much less, 2940 CFU/ml to 3880 CFU/ml and 2850 CFU/ml to 3230 CFU/ml respectively (Figures 5.20). This decrease in the increasing trend is explained by the effect of the increase in the oil content of the synthetic produced water on the aerobic metabolic activity. Figure 5.20 show a comparison between the bacterial colony plate count increasing trends in the three stages of the operation of the hybrid system.

Nevertheless, colony plate count does not take into account the population of unculturable bacteria. Measurement of total protein does not suffer from this disadvantage and has previously been used for quantification of halophilic bacterial populations (Dastgheib et al., 2011). The only potential drawback with the use of total protein data as a measure of bacterial population is the fact that it includes both the cellular protein as well as the Protein Soluble Microbial Product (SMPp). This is not a problem if the major part of SMPp is utilization associated products (UAB), which correlates with growth, as opposed to biomass associated products (BAP) which mainly includes compounds formed as a result of cell lysis (Jiang et al., 2008) [33].

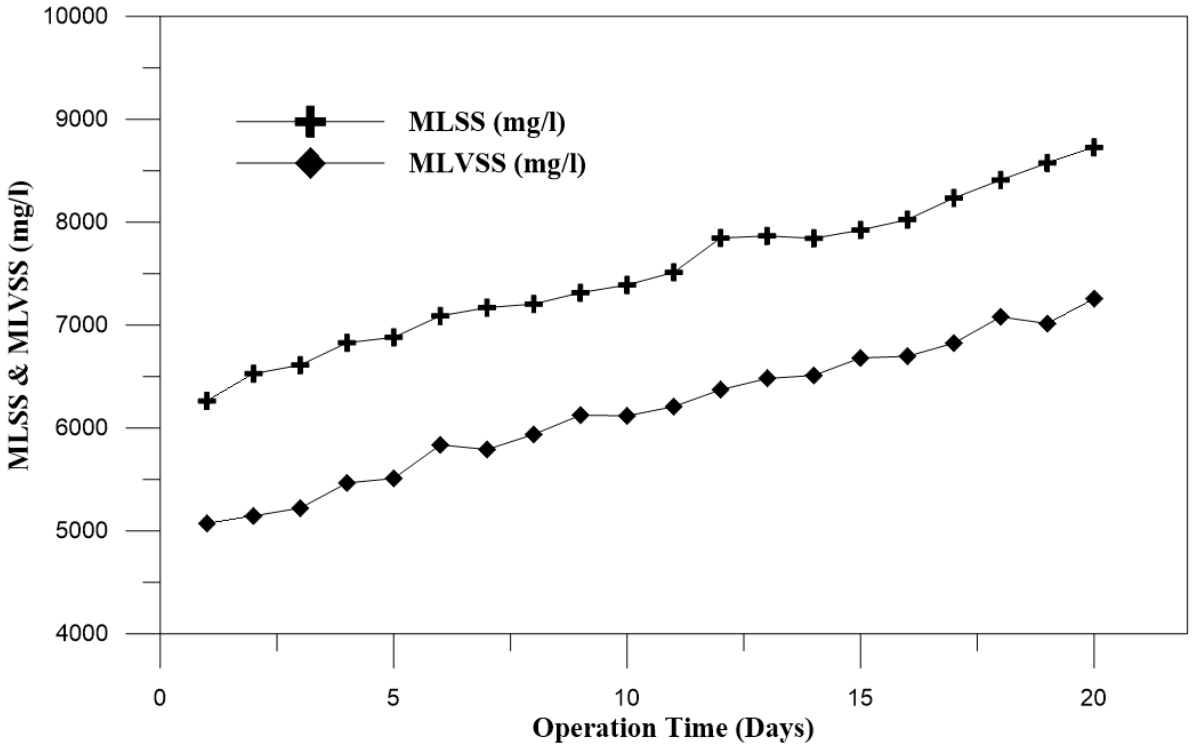


Figure 5.16 MLSS & MLVSS vs Operation Time (Days) – Stage1 – Hybrid System

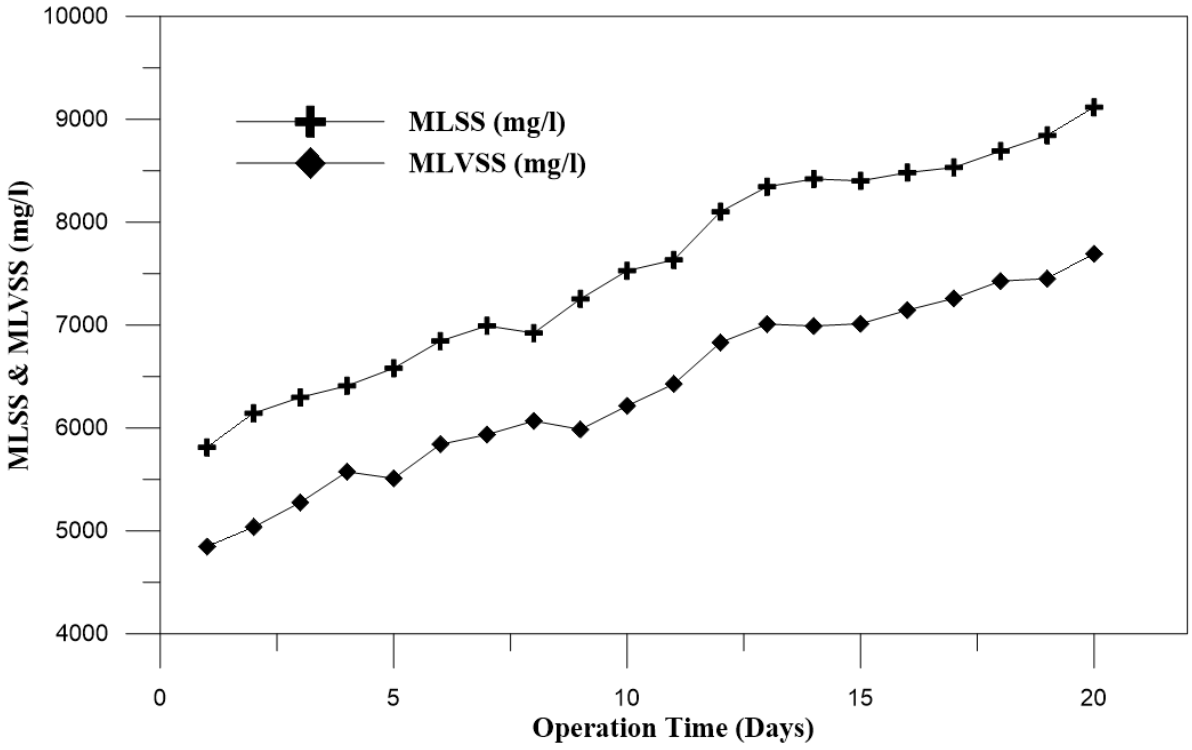


Figure 5.17 MLSS & MLVSS vs Operation Time (Days) – Stage2 – Hybrid System

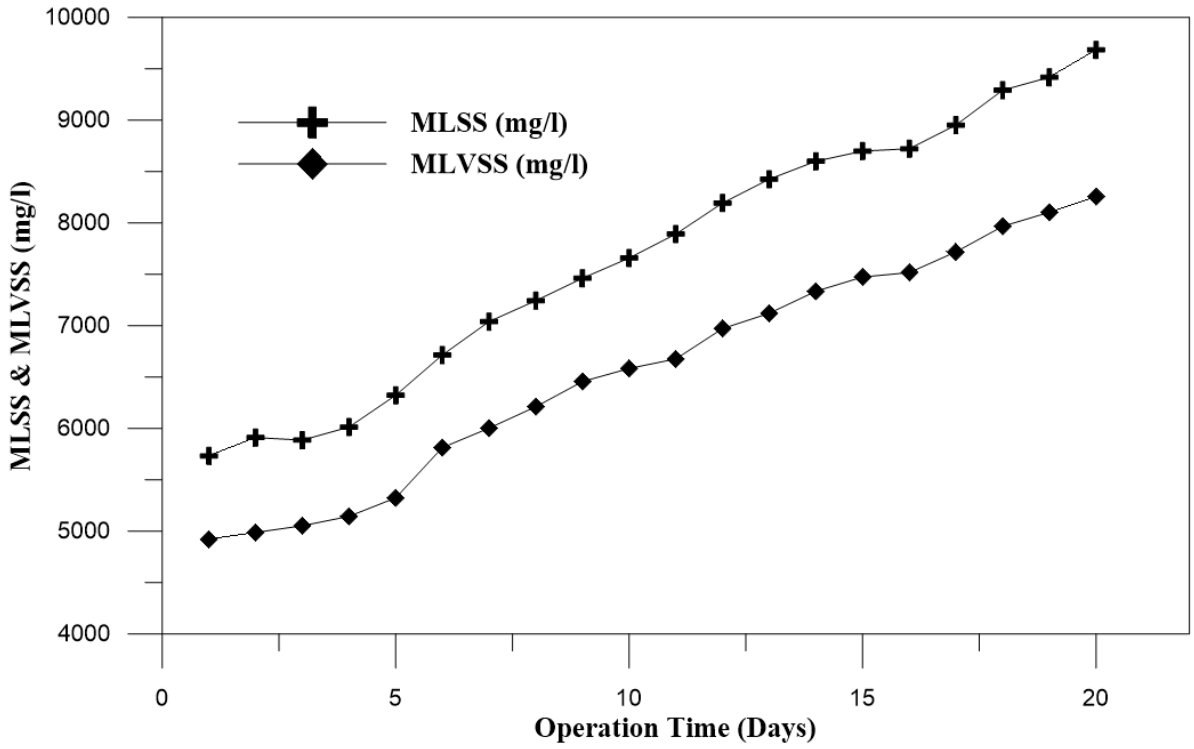


Figure 5.18 MLSS & MLVSS vs Operation Time (Days) – Stage3 – Hybrid System

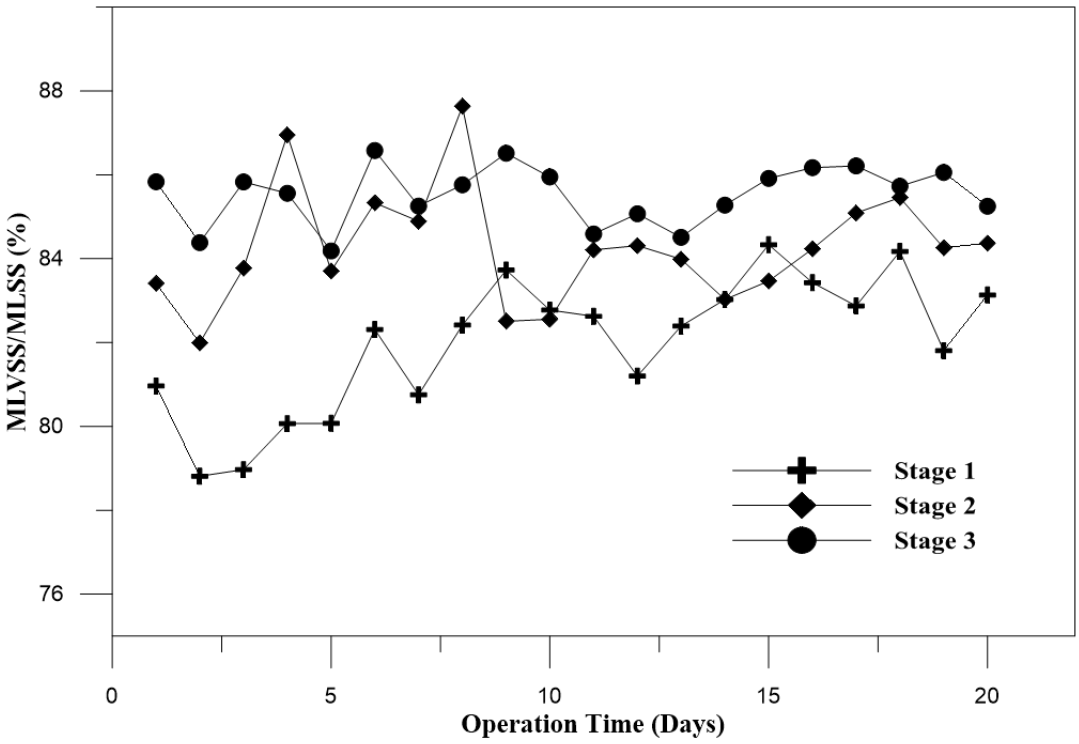


Figure 5.19 MLSS/MLVSS (%) vs Operation Time (Days) – Stage 1, 2 and 3 – Hybrid System

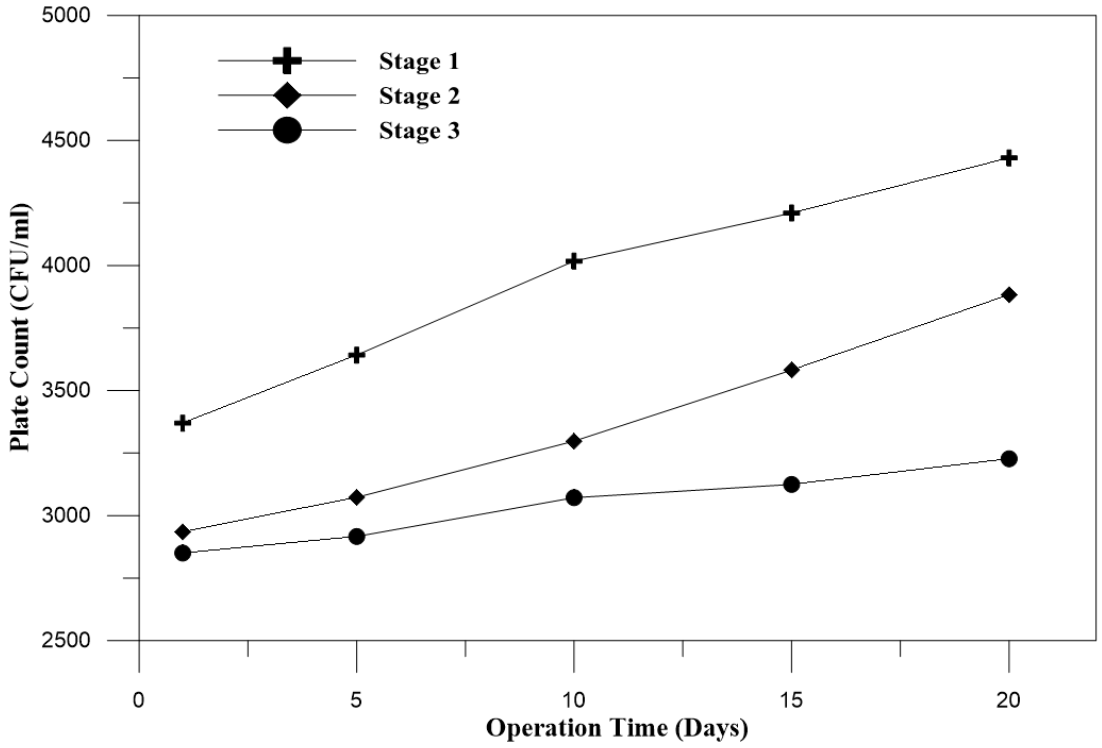


Figure 5.20 Plate Count (CFU/ml) vs Operation Time (Days) – Stage 1, 2 and 3 – Hybrid System

5.3.4 COD Removal

The performance of the hybrid system was studied to assess the ability and stability of the system to provide the required COD removal. The performance of the hybrid system at the three different stages are presented. At the beginning of the study, the MLVSS concentration was chosen to be 5000 mg/l. The liquor was brown in color and made up of dispersed non flocculent particles.

Figure 5.21 represents the COD removal efficiency of the hybrid system during the three different stages. The COD removal efficiency ranged between 91% and 97% with an average of 94%.

Generally the performance was impressive with changing the influent oil content from 100 to 200 mg/L. The trend of change of COD removal efficiencies during the operation of the hybrid system during the three stages are presented in Figure 5.21. The maximum COD removal efficiency was achieved during stage 1 with an average removal efficiency of 96%. Whereas, stages 2 and 3 achieved 93% and 92% COD removal efficiencies respectively. The decrease in the removal efficiencies of COD between the three stages showed that the hybrid system in general and the membrane bioreactor performance specifically is effected by the increase in the oil content in the synthetic produced water among the three stages. Moreover, the decrease in the removal efficiency within the stage in stage 2 and 3 of the operation show that the membrane bioreactor performance is effected by the accumulation of the oil in the mixed liquor.

The decrease of the COD removal efficiency between the three stages and within stage 2 and 3 can be explained by the inhibitory effect of the increase in oil concentration in the synthetic produced water and the accumulation of oil in the mixed liquor on the aerobic metabolic activity and hence the growth rate of the halo-philic bacterial consortium as evidenced by the bacterial colony plate count presented in section 5.3.3. This can be further attributed to lower microbial diversity and increase in hydrophilicity of the cell surfaces of halo-philic bacteria as well as reduced solubility of hydrocarbons with increase in the oil concentration.

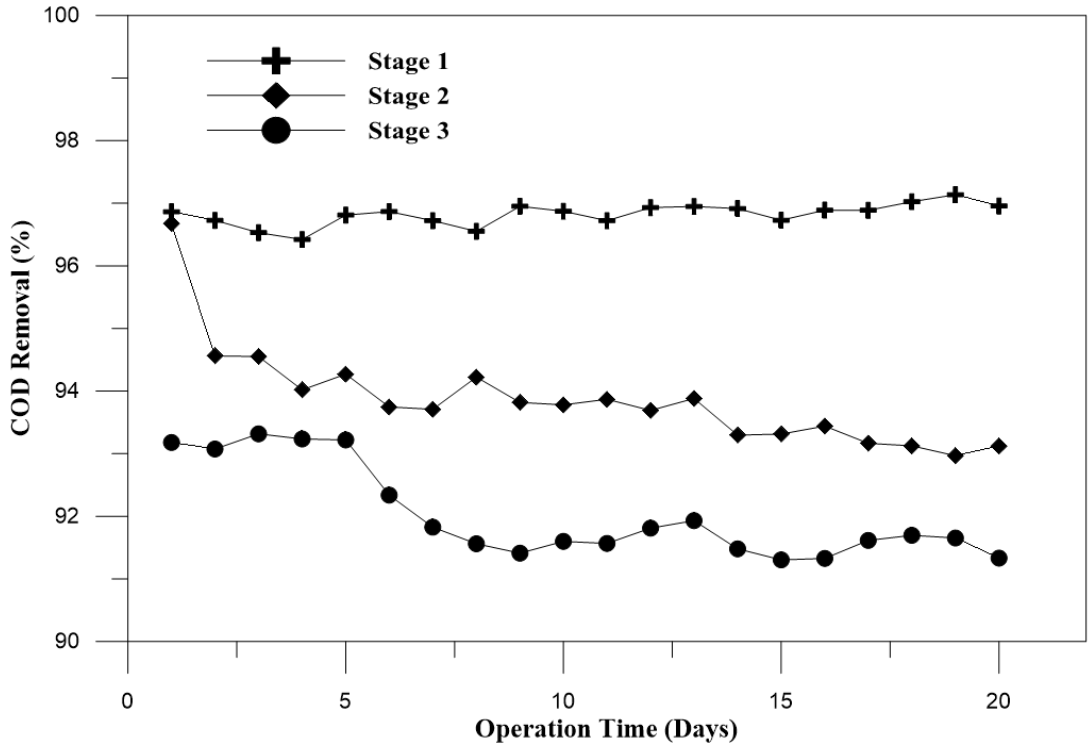


Figure 5.21 COD Removal (%) vs Operation Time (Days) – Stage 1, 2 and 3 – Hybrid System

5.3.5 Oil & Grease Removal

During the hybrid system 60 days of operation, the oil & grease in the effluent increased from an average of 6.04 mg/L with 94% average removal efficiency in stage 1 to a an average of 33.28 mg/L with average removal efficiency of 83% in stage 3 (Figure 5.22). This increase of the oil & grease in the effluent and the decrease of the removal efficiency is a result of the increase in the oil content in the synthetic produced water used among the three stages from 100 mg/L to 200 mg/L. Furthermore, the accumulation of the oil & grease in the mixed liquor decreased the growth rate of the halo-philic bacterial consortium as showed in section 5.3.3 and hence decreased the removal efficiency of the system between the three stages and during the same stage as in stage 2 and 3. Figure 5.22 compare the oil & grease removal efficiency among the three stages of the operation of the hybrid system. (Sharghi et al., 2014) [49] in their study of treating a hypersaline produced water employing a moderately halophilic bacterial consortium, showed a similar decreasing trend with time in the oil & grease removal efficiency. Their results showed a decrease from 94% to 84% between day 1 to day 85 of the experiment. The decrease of oil & grease removal can be explained by the inhibitory effect of the increase of the oil content on the aerobic metabolic activity and hence on the growth rate of the halophilic bacterial consortium as evidenced in section 5.3.3.

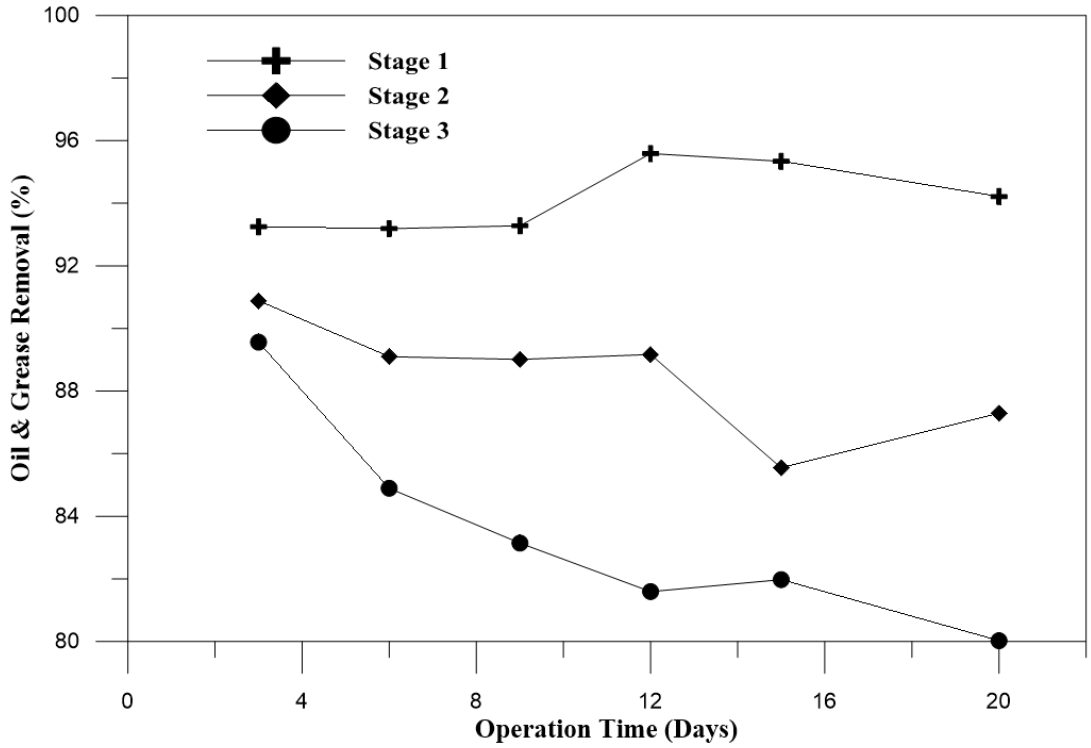


Figure 5.22 Oil & Grease Removal (%) vs Operation Time (Days) – Stage 1, 2 and 3 – Hybrid System

5.3.6 Particle Size Distribution

Particle size distribution analysis presented in Figures 5.23, 5.24 and 5.25 show an increase in the mean flocs size throughout the three stages with the increase in the oil concentration from 100 mg/L to 200 mg/L. The particle size distribution analysis show a mean value of 15.63 µm for stage 1, a mean value of 15.96 µm for stage two and a large increase in the mean value of stage 3 with 22.72 µm.

The particle size distribution can correspond to both dispersed bacteria and emulsified oil particles in the mixed liquor. Whereas, due to the results obtained from the MLSS & MLVSS and plate count results (section 5.3.3), the increase in the mean particle size is contributed to the accumulation of the oil in the mixed liquor. (Shargi et. al 2014) [49], showed a lowering of the mean floc size with increase in NaCl concentration from 100 g/L (D0.5 = 15.9 µm) to 250 g/L (D0.5 = 11.2 µm). Also, their results showed that the particle size distribution curves at 200 and 250 g/L NaCl had a higher left hand area compared to the corresponding curves at lower salt concentrations; since the left hand area of the particle size distribution curve corresponds to particles with sizes that potentially correspond to both dispersed bacteria and emulsified oil particles this increase can indicate the increase in the population of either or both of these particle types.

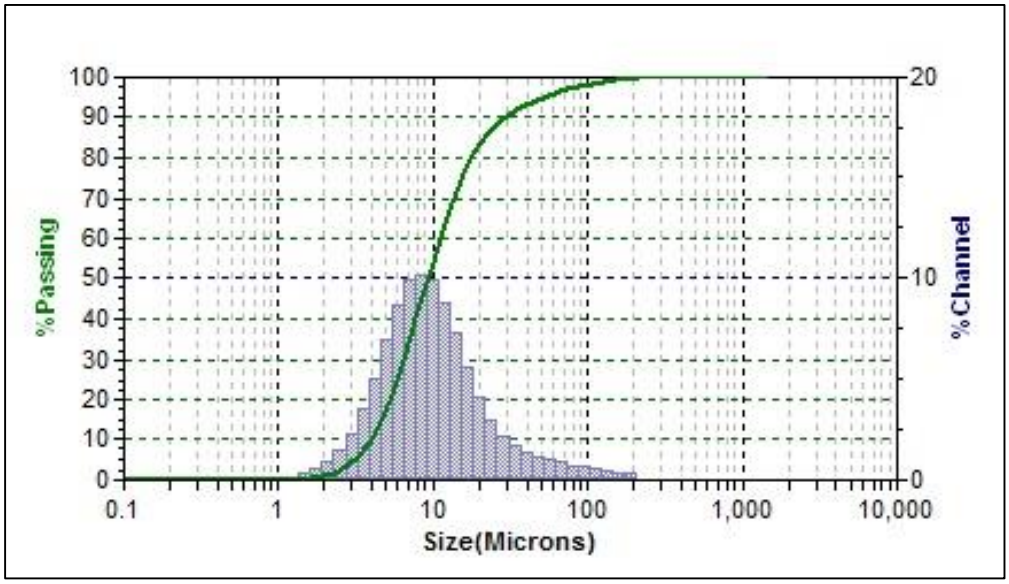


Figure 5.23 Particle Size Distibution – Stage1 – Hybrid System

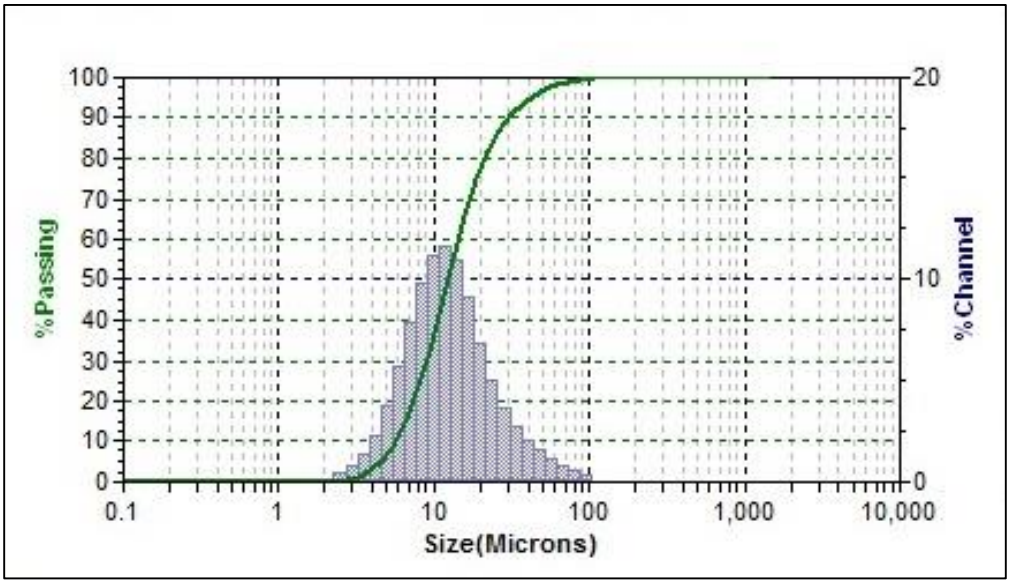


Figure 5.24 Particle Size Distibution – Stage2 – Hybrid System

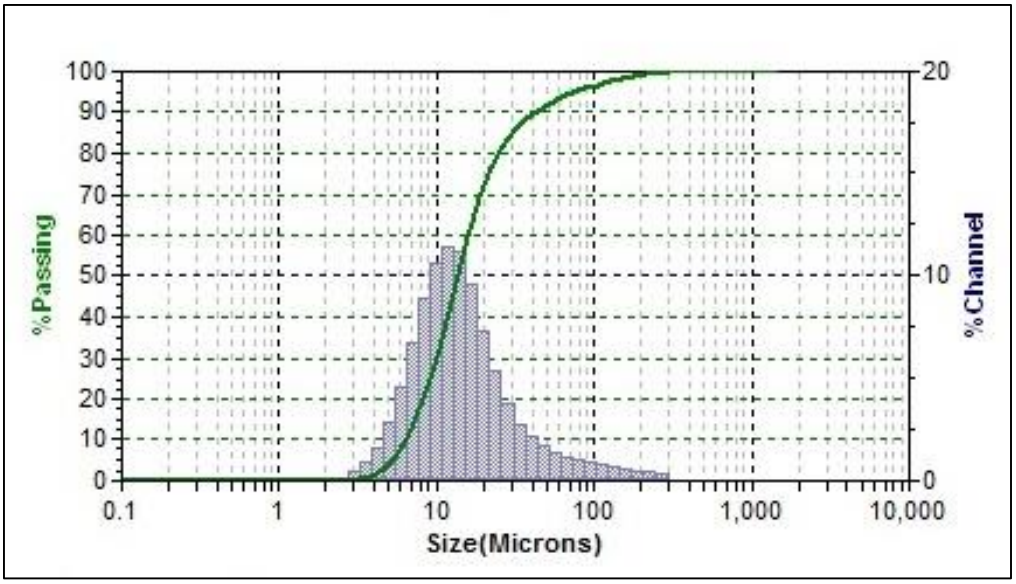


Figure 5.25 Particle Size Distibution – Stage3 – Hybrid System

CHAPTER 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 General Conclusions

- In the electrocoagulation experiments, the increase in both the electrolysis time and the current density caused an increase in the COD and Turbidity removal efficiency. Furthermore, Aluminum electrodes achieved a much higher removal efficiency than Stainless Steel electrodes.
- The membrane performance in the preliminary experiments affected by the increase in flux. At a flux of 20 L/hr.m^2 membrane fouling occurred after 24 hours of operation and the membrane needed to be backwashed for 10 minutes every 24 hours.
- In stage 1 of the hybrid system, the MLSS and the MLVSS reached 8727 mg/L and 7255 mg/L respectively. The bacterial plate count increased from 3370 to 4430 CFU/ml. Moreover, an average COD removal efficiency of 96.82% and oil & grease removal efficiency of 94.14% were achieved.
- In stage 2 of the hybrid system, the MLSS and the MLVSS reached 9117 mg/L and 7692 mg/L respectively. The bacterial plate count increased from 2940 to 3880 CFU/ml. Moreover, an average COD removal efficiency of 93.86% and oil & grease removal efficiency of 88.50% were achieved.
- In stage 3 of the hybrid system, the MLSS and the MLVSS reached 9683 mg/L and 8255 mg/L respectively. The bacterial plate count increased from 2850 to 3230

CFU/ml. Moreover, an average COD removal efficiency of 92.02% and oil & grease removal efficiency of 83.53% were achieved.

- The transmembrane pressure during each phase of the hybrid system remained in the range of 0.5 to 4.0 psi and the need for membrane washing never arose.
- The hybrid system containing the electrocoagulation unit and the membrane bioreactor containing a moderately halophilic bacterial consortium showed a good performance in treating the synthetic produced water at the three different oil content.

6.2 Recommendations

The following points are in need of further research:

- More electrodes material in the electrocoagulation cell and different material combinations.
- The effect of running the membrane at the maximum operating pressure.
- The effect of changing the initial MLVSS of the membrane bioreactor.
- The higher oil content that the hybrid system can withstand.

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APPENDIX A

RAW DATA

EC Exp. COD Removal Efficiency – Alum. Electrodes - Current Density = 5 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1652.21	1.95
15	1601.76	4.94
30	1422.82	15.56
60	1249.06	25.87
90	1115.90	33.77
120	1062.44	36.95

EC Exp. COD Removal Efficiency – Alum. Electrodes - Current Density = 10 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1647.26	2.24
15	1578.32	6.33
30	1305.32	22.53
60	1161.19	31.09
90	978.11	41.95
120	838.45	50.24

EC Exp. COD Removal Efficiency – Alum. Electrodes - Current Density = 30 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1647.26	2.24
15	1578.32	6.33
30	1305.32	22.53
60	1161.19	31.09
90	978.11	41.95
120	838.45	50.24

EC Exp. COD Removal Efficiency – S.S. Electrodes - Current Density = 5 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1678.31	0.39
15	1639.29	2.71
30	1526.16	9.42
60	1489.38	11.61
90	1361.01	19.23
120	1320.24	21.65

EC Exp. COD Removal Efficiency – S.S. Electrodes - Current Density = 10 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1661.82	1.38
15	1602.23	4.91
30	1491.02	11.51
60	1325.25	21.35
90	1274.14	24.38
120	1243.56	26.19

EC Exp. COD Removal Efficiency – S.S. Electrodes - Current Density = 30 mA/cm^2

Time (min.)	COD Concentration (mg/L)	COD Removal Efficiency (%)
0	1685.00	0.00
5	1642.02	2.55
15	1572.12	6.69
30	1292.25	23.30
60	1105.47	34.39
90	949.51	43.65
120	898.22	46.69

EC Exp. Turbidity Removal Effic. – Aluminum Electrodes - Current Density = 5 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	123.31	4.26
15	114.29	11.27
30	110.50	14.21
60	105.21	18.32
90	99.40	22.83
120	91.10	29.27

EC Exp. Turbidity Removal Effic. – Alum. Electrodes - Current Density = 10 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	119.23	7.43
15	105.23	18.30
30	98.10	23.84
60	91.60	28.88
90	85.65	33.50
120	81.23	36.93

EC Exp. Turbidity Removal Effic. – Alum. Electrodes - Current Density = 30 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	114.50	11.10
15	91.20	29.19
30	85.91	33.30
60	72.12	44.01
90	68.40	46.89
120	62.20	51.71

EC Exp. Turbidity Removal Effic. – S.S. Electrodes - Current Density = 5 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	128.02	0.61
15	122.40	4.97
30	116.20	9.78
60	111.80	13.20
90	108.40	15.84
120	102.30	20.57

EC Exp. Turbidity Removal Effic. – S.S. Electrodes - Current Density = 10 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	126.90	1.48
15	121.41	5.74
30	112.20	12.89
60	105.60	18.01
90	98.34	23.65
120	95.72	25.68

EC Exp. Turbidity Removal Effic. – S.S. Electrodes - Current Density = 30 mA/cm^2

Time (min.)	Turbidity (NTU)	Turbidity Removal Efficiency (%)
0	128.80	0.00
5	124.03	3.70
15	120.52	6.43
30	109.35	15.10
60	105.20	18.32
90	95.34	25.98
120	93.80	27.17

Membrane Fouling Performance Experiment 1

Exp. 1 – (Flux = 10 L/hr.m^2)		
Time (hours)	Pressure (psi)	Actual Flux (L/hr.m^2)
0	0	10.00
0.5	0.5	10.00
1	0.5	10.00
2	0.5	10.00
3	0.5	10.00
6	0.5	10.00
12	0.5	10.00
24	0.5	10.00
36	0.75	10.00
48	1.0	9.60 (Adjusted)
60	1.0	10.00
72	1.0	10.00
84	1.0	10.00
96	1.0	10.00

Membrane Fouling Performance Experiment 2

Exp. 2 – (Flux = 11.43 L/hr.m^2)		
Time (hours)	Pressure (psi)	Actual Flux (L/hr.m^2)
0	0	11.43
0.5	0.5	11.10 (Adjusted)
1	0.5	11.43
2	1.0	11.43
3	1.5	11.43
6	1.5	11.43
12	2.5	11.43
24	2.5	11.43
36	3.0	11.03 (Adjusted)
48	3.5	11.43
60	3.5	11.43
72	3.5	11.43
84	3.5	11.43
96	3.5	11.43

Membrane Fouling Performance Experiment 3

Exp. 3 – (Flux = 13.33 L/hr.m^2)		
Time (hours)	Pressure (psi)	Actual Flux (L/hr.m^2)
0	0	13.33
0.5	1.0	13.33
1	1.0	13.33
2	2.0	12.90 (Adjusted)
3	2.5	13.30
6	3.5	13.30
12	4.0	12.90 (Adjusted)
24	4.0	13.30
36	4.0	13.30
48	4.0	13.30
60	4.0	13.30
72	4.0	13.30
84	4.0	13.30
96	4.0	13.30

Membrane Fouling Performance Experiment 4

Exp. 4 – (Flux = 16.00 L/hr.m^2)		
Time (hours)	Pressure (psi)	Actual Flux (L/hr.m^2)
0	0	16.00
0.5	1.0	16.00
1	1.5	15.85 (Adjusted)
2	1.5	16.00
3	2.5	15.60 (Adjusted)
6	2.5	16.00
12	2.5	16.00
24	3.0	16.00
36	4.0	15.60 (Adjusted)
48	4.0	16.00
60	4.0	16.00
72	4.5	15.85 (Adjusted)
84	4.5	16.00
96	4.5	16.00

Membrane Fouling Performance Experiment 5

Exp. 5 – (Flux = 20 L/hr.m^2)		
Time (hours)	Pressure (psi)	Actual Flux (L/hr.m^2)
0	0	20.00
0.5	3.0	19.50 (Adjusted)
1	4.0	19.65 (Adjusted)
2	5.0	19.20 (Adjusted)
3	5.0	20.00
6	5.0	20.00
12	6.0	19.65 (Adjusted)
24	7.0 (Adjusted – Backwash No.1)	19.13 (Adjusted)
36	6.0	20.00
48	7.0 (Adjusted – Backwash No.2)	19.50 (Adjusted)
60	6.0	20.00
72	7.0 (Adjusted – Backwash No.3)	19.50 (Adjusted)
84	6.0	20.00
96	7.0 (Adjusted – Backwash No.4)	19.50 (Adjusted)
108	6.0	20.00
120	7.0	19.50

After Backwash No.1 (at 24 hours)		
Time (minutes)	Pressure (psi)	Actual Flux (L/hr.m2)
15	3.0	19.50 (Adjusted)
30	3.5	20.00
45	4.0	19.65 (Adjusted)
60	4.0	20.00
90	5.0	19.65 (Adjusted)
120	6.0	19.50 (Adjusted)

After Backwash No.2 (at 48 hours)		
Time (minutes)	Pressure (psi)	Actual Flux (L/hr.m2)
15	3.5	19.50 (Adjusted)
30	3.5	20.00
45	4.0	20.00
60	4.5	20.00
90	5.0	19.65 (Adjusted)
120	6.0	19.65 (Adjusted)

After Backwash No.3 (at 72 hours)		
Time (minutes)	Pressure (psi)	Actual Flux (L/hr.m2)
15	3.5	19.50 (Adjusted)
30	3.5	20.00
45	4.0	20.00
60	4.5	20.00
90	5.0	19.65 (Adjusted)
120	6.0	19.65 (Adjusted)

After Backwash No.4 (at 96 hours)		
Time (minutes)	Pressure (psi)	Actual Flux (L/hr.m2)
15	3.5	19.50 (Adjusted)
30	4.0	20.00
45	4.0	20.00
60	4.5	20.00
90	5.5	19.50 (Adjusted)
120	6.0	19.50 (Adjusted)

Hybrid System – Actual Flux and Transmembrane Pressure – Stage1

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
0	12	0
0.5	11.80 (Adjusted)	0.5
1	12	0.5
2	12	1
3	12	1.5
6	12	1.5
12	11.55 (Adjusted)	2.5
24	12	3.5
36	12	3.5
48	12	3.5
60	12	3.5
72	12	3.5
84	12	3.5
96	12	3.5
108	12	3.5
120	12	3.5
132	12	3.5
144	12	3.5
156	12	3.5
168	12	3.5
180	11.55 (Adjusted)	4.0
192	12	4.0
204	12	4.0
228	12	4.0
240	12	4.0
252	12	4.0
264	12	4.0
276	12	4.0
288	12	4.0
300	12	4.0
312	12	4.0
324	12	4.0
336	12	4.0
348	12	4.0
360	12	4.0
372	12	4.0
384	12	4.0
396	12	4.0
408	12	4.0

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
420	12	4.0
432	12	4.0
444	12	4.0
456	12	4.0
468	12	4.0
480	12	4.0

Hybrid System – Actual Flux and Transmembrane Pressure – Stage2

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
0	12	0
0.5	11.75 (Adjusted)	0.5
1	12	1
2	12	1.5
3	12	1.5
6	12	1.5
12	11.25 (Adjusted)	3.5
24	12	3.5
36	12	3.5
48	12	3.5
60	12	3.5
72	12	3.5
84	12	3.5
96	12	3.5
108	12	3.5
120	12	3.5
132	11.25 (Adjusted)	4.0
144	12	4.0
156	12	4.0
168	12	4.0
180	12	4.0
192	12	4.0
204	12	4.0
228	12	4.0
240	12	4.0
252	12	4.0
264	12	4.0
276	12	4.0
288	12	4.0
300	12	4.0
312	12	4.0
324	12	4.0
336	12	4.0
348	12	4.0
360	11.40 (Adjusted)	4.0
372	12	4.0
384	12	4.0
396	12	4.0
408	12	4.0

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
420	12	4.0
432	12	4.0
444	12	4.0
456	12	4.0
468	12	4.0
480	12	4.0

Hybrid System – Actual Flux and Transmembrane Pressure – Stage3

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
0	12	0
0.5	11.25 (Adjusted)	0.5
1	12	1
2	11.50 (Adjusted)	1.5
3	12	1.5
6	12	1.5
12	11.25 (Adjusted)	3.5
24	12	3.5
36	12	3.5
48	12	3.5
60	12	3.5
72	12	3.5
84	12	3.5
96	12	3.5
108	12	3.5
120	12	3.5
132	12	3.5
144	12	3.5
156	12	3.5
168	11.25 (Adjusted)	4.0
180	12	4.0
192	12	4.0
204	12	4.0
228	12	4.0
240	11.25 (Adjusted)	4.0
252	12	4.0
264	12	4.0
276	12	4.0
288	12	4.0
300	12	4.0
312	12	4.0
324	12	4.0
336	12	4.0
348	12	4.0
360	12	4.0
372	12	4.0
384	12	4.0
396	12	4.0

Time (hours)	Actual Flux (L/h.m2)	Pressure (psi)
408	12	4.0
420	11.40 (Adjusted)	4.0
432	12	4.0
444	12	4.0
456	12	4.0
468	12	4.0
480	12	4.0

Hybrid System – MLSS and MLVSS – Stage1

Day	MLSS (mg/L)	MLVSS (mg/L)	MLVSS/MLSS (%)
1	6262	5070	80.96
2	6528	5145	78.81
3	6610	5220	78.97
4	6827	5466	80.06
5	6880	5509	80.07
6	7090	5836	82.31
7	7170	5790	80.75
8	7205	5938	82.41
9	7314	6124	83.73
10	7390	6117	82.77
11	7512	6207	82.63
12	7846	6371	81.20
13	7866	6481	82.39
14	7842	6511	83.03
15	7922	6681	84.33
16	8025	6695	83.43
17	8234	6824	82.88
18	8410	7079	84.17
19	8574	7014	81.81
20	8727	7255	83.13

Hybrid System – MLSS and MLVSS – Stage2

Day	MLSS (mg/L)	MLVSS (mg/L)	MLVSS/MLSS (%)
1	5810	4846	83.41
2	6142	5036	81.99
3	6298	5276	83.77
4	6409	5573	86.96
5	6580	5508	83.71
6	6844	5840	85.33
7	6992	5936	84.90
8	6923	6067	87.64
9	7254	5985	82.51
10	7527	6214	82.56
11	7633	6428	84.21
12	8101	6830	84.31
13	8345	7009	83.99
14	8419	6990	83.03
15	8402	7013	83.47
16	8481	7144	84.24
17	8531	7259	85.09
18	8692	7428	85.46
19	8843	7451	84.26
20	9117	7692	84.37

Hybrid System – MLSS and MLVSS – Stage3

Day	MLSS (mg/L)	MLVSS (mg/L)	MLVSS/MLSS (%)
1	5732	4920	85.83
2	5910	4987	84.38
3	5886	5052	85.83
4	6012	5144	85.56
5	6323	5323	84.18
6	6714	5813	86.58
7	7040	6002	85.26
8	7242	6211	85.76
9	7461	6455	86.52
10	7658	6582	85.95
11	7891	6675	84.59
12	8193	6970	85.07
13	8424	7119	84.51
14	8600	7334	85.28
15	8698	7473	85.92
16	8722	7516	86.17
17	8951	7717	86.21
18	9292	7966	85.73
19	9416	8103	86.06
20	9683	8255	85.25

Hybrid System – COD Removal Efficiency and Effluent – Stage1

Day	Effluent COD (mg/L)	Removal %	Influent COD (mg/L)
1	52.66	96.87	1680.00
2	55.00	96.73	1680.00
3	58.27	96.53	1680.00
4	60.14	96.42	1680.00
5	52.73	96.81	1654.00
6	51.80	96.87	1654.00
7	54.20	96.72	1654.00
8	57.06	96.55	1654.00
9	50.42	96.95	1654.00
10	51.72	96.87	1654.00
11	54.20	96.72	1654.00
12	50.77	96.93	1654.00
13	50.48	96.95	1654.00
14	51.03	96.91	1654.00
15	54.80	96.73	1675.00
16	52.09	96.89	1675.00
17	52.14	96.89	1675.00
18	50.11	97.03	1685.00
19	48.25	97.14	1685.00
20	51.30	96.96	1685.00

Hybrid System – COD Removal Efficiency and Effluent – Stage2

Day	Effluent COD (mg/L)	Removal %	Influent COD (mg/L)
1	58.20	96.68	1751.00
2	95.22	94.56	1751.00
3	95.41	94.55	1751.00
4	102.50	94.02	1714.00
5	98.20	94.27	1714.00
6	107.19	93.75	1714.00
7	107.84	93.71	1714.00
8	103.00	94.22	1783.00
9	110.20	93.82	1783.00
10	110.90	93.78	1783.00
11	109.30	93.87	1783.00
12	112.50	93.69	1783.00
13	109.10	93.88	1783.00
14	115.30	93.30	1720.05
15	115.00	93.31	1720.05
16	112.80	93.44	1720.05
17	117.55	93.17	1720.05
18	118.91	93.12	1729.50
19	121.64	92.97	1729.50
20	118.95	93.12	1729.50

Hybrid System – COD Removal Efficiency and Effluent – Stage3

Day	Effluent COD (mg/L)	Removal %	Influent COD (mg/L)
1	121.03	93.18	1774.30
2	122.90	93.07	1774.30
3	118.60	93.32	1774.30
4	120.05	93.23	1774.30
5	120.30	93.22	1774.30
6	138.10	92.34	1802.51
7	147.31	91.83	1802.51
8	152.10	91.56	1802.51
9	154.82	91.41	1802.51
10	151.44	91.60	1802.51
11	151.69	91.57	1798.72
12	147.30	91.81	1798.72
13	145.15	91.93	1798.72
14	153.25	91.48	1798.72
15	156.41	91.30	1798.72
16	156.02	91.33	1798.72
17	150.83	91.61	1809.81
18	149.36	91.70	1809.81
19	150.13	91.65	1809.81
20	155.93	91.33	1809.81

Bacterial Colony Plate Count – Stage1

Day	Sample						Min.	Max.	Average	Std. Deviation
	1	2	3	4	5	6				
1	3400	3560	3050	3480	3310	3420	3050	3560	3370	177.54
5	3470	3700	3520	3670	3810	3680	3470	3810	3642	125.13
10	3920	4080	4110	3960			3920	4110	4018	91.79
15	3810	4310	4220	4370	4160	4390	3810	4390	4210	214.76
20	4210	4480	4360	4570	4670	4290	4210	4670	4430	174.70

Bacterial Colony Plate Count – Stage2

Day	Sample						Min.	Max.	Average	Std. Deviation
	1	2	3	4	5	6				
1	2960	3090	3250	2850	2820	2640	2640	3250	2935	215.10
5	3020	3180	2970	3390	3000	2880	2880	3390	3073	183.27
10	3350	3510	3110	3160	3360		3110	3510	3298	162.70
15	3390	3740	3540	3710	3490	3620	3390	3740	3582	134.08
20	3890	3910	3720	3990	3850	3940	3720	3990	3883	92.88

Bacterial Colony Plate Count – Stage3

Day	Sample						Min.	Max.	Average	Std. Deviation
	1	2	3	4	5	6				
1	2820	2740	2950	2890			2740	2950	2850	90.55
5	2980	2790	2800	2940	2970	3020	2790	3020	2917	97.71
10	3330	3130	2980	2910	3010		2910	3330	3072	164.68
15	3210	3210	2960	3170	3110	3090	2960	3210	3125	95.03
20	3320	3290	3110	3190			3110	3320	3228	96.05

Hybrid System – Oil & Grease Removal Efficiency and Effluent – Stage1

Day	Oil & Grease (mg/L)	Influent Oil & Grease (mg/L)	Removal %
3	6.98	103.40	93.25
6	7.04	103.40	93.19
9	6.95	103.40	93.28
12	4.56	103.40	95.59
15	4.82	103.40	95.34
20	5.91	102.11	94.21

Hybrid System – Oil & Grease Removal Efficiency and Effluent – Stage2

Day	Oil & Grease (mg/L)	Influent Oil & Grease (mg/L)	Removal %
3	14.25	156.23	90.88
6	16.35	150.00	89.10
9	16.10	146.50	89.01
12	16.55	152.80	89.17
15	22.08	152.80	85.55
20	19.72	155.15	87.29

Hybrid System – Oil & Grease Removal Efficiency and Effluent – Stage3

Day	Oil & Grease (mg/L)	Influent Oil & Grease (mg/L)	Removal %
3	20.61	197.45	89.56
6	29.50	195.30	84.90
9	32.92	195.30	83.14
12	38.69	210.22	81.60
15	37.33	207.11	81.98
20	40.63	203.41	80.03

APPENDIX B

INSTALLATION & OPERATING MANUAL

for

ZeeWeed - 1 (ZW-1) Bench Test Unit

Revision: 1.1

ZENON Environmental Systems Inc.
3239 Dundas Street West
Oakville, Ontario
L6M 4B2
phone: (905) 465-3030
fax: (905) 465-3050

July 2001
Version: 010628HB

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SYSTEM DESCRIPTION

The ZeeWeed®-1 bench test unit is a simple, compact ZW membrane filtration system that can be used to generate limited preliminary performance data prior to further evaluation or pilot scale testing. It uses the same membrane as the full-scale ZeeWeed®-500 modules and hence the effluent quality is representative of full-scale modules. As importantly, the unit can be used to evaluate membrane fouling and cleaning for user's process application. However, as fibers are much shorter and tighter (compared to larger modules that have substantial slack to permit fibre movement, and as aeration pattern is different, a ZW-1 module cannot be used to study filtration variables such as flux, pressure and energy requirements. Also care should be taken in interpreting fouling data if excessive solids build up in the module occurs during operation

The ZW-1 bench system can be used in both batch and continuous modes. The system can be configured either as a ZenoGem activated sludge system or as a direct filtration system.

Zenon supplies only the membrane module and assembly/operating instructions. The customer is responsible for assembling a simple system using readily available parts. A 2 to 20 liter beaker, bucket or cylinder serves as the process tank with overflow high enough for the membranes to be fully submerged. The ZW-1 module can be positioned in the beaker/bucket by the use of a retort stand and clamp arrangement. Although backpulsing is not usually done with this system, if required, a small backpulse tank is placed on the permeate stream with the permeate inlet line being fully submerged. Typically, the process pump and blower, if used, will be located on a table adjacent to the process tank. 1/4" and 3/8" polyethylene tubing is used for the majority of the connections. Instrumentation consists of a digital (battery powered) pressure indicator, an air flow indicator (rotameter) and a temperature indicator in the process tank. Flows are measured manually with a graduated cylinder and stopwatch. Equipment and sources are specified in Appendix A.

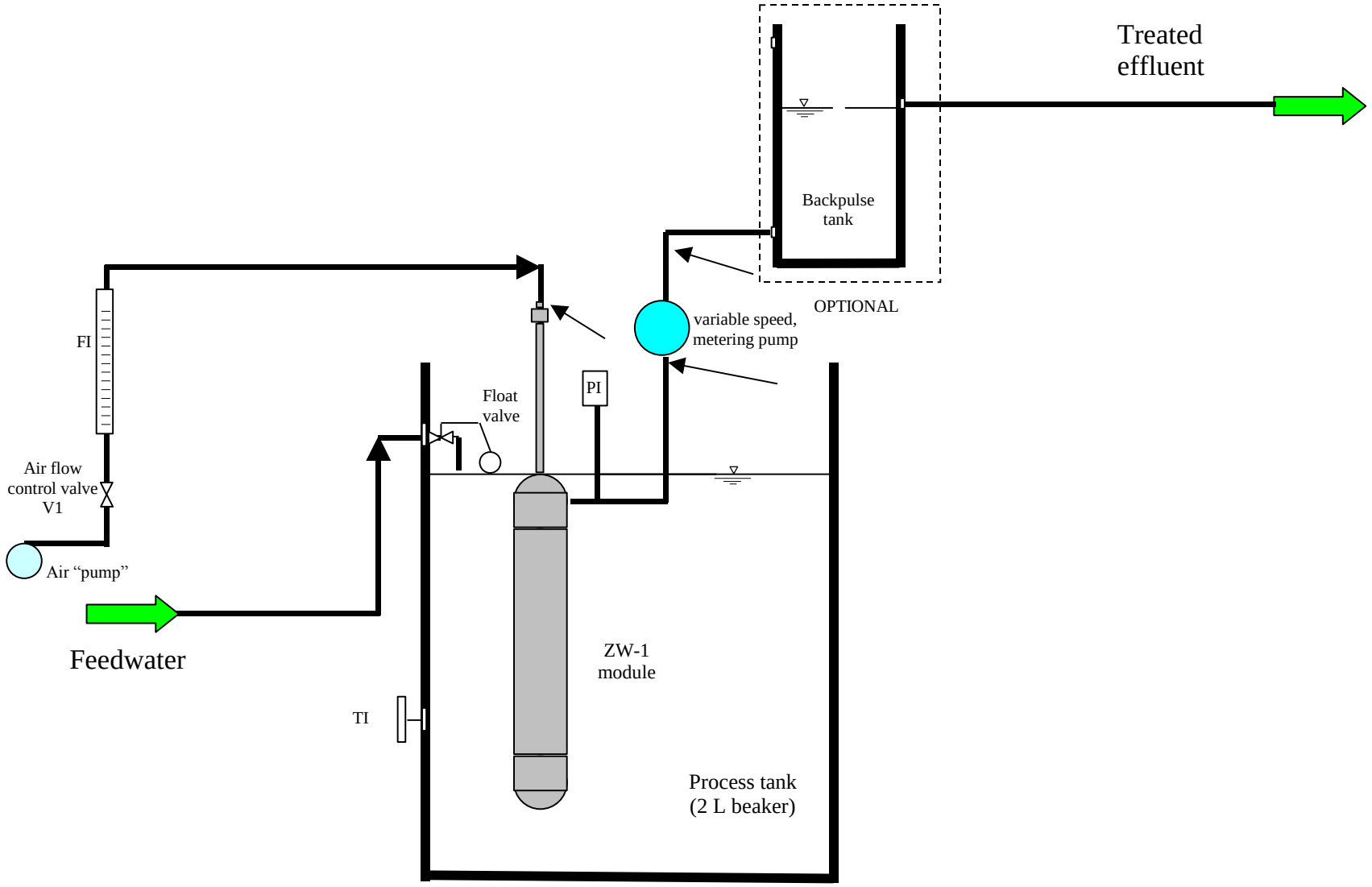

Figure 1

ZW-1 P & ID

COMPONENT DESCRIPTIONS

The primary components of the system are described in the following sections. All equipment other than the ZW-1 membrane module is customer supplied. Procurement information on main components is presented in appendix A.

PROCESS PUMP

The process pump is a variable speed positive displacement pump (peristaltic, piston/diaphragm, or gear) with a flow range of about 10 to 150 ml/min. We recommend a micro-gear pump, however, an equivalent in a peristaltic or piston/diaphragm pump can be used. A reversible flow version is required for backwash if used.

PROCESS TANK

The tank is a 2 to 4 liter polyethylene graduated cylinder or beaker with sufficient depth. The feed flow will be controlled by the level of the tank through a float valve V2. The float valve may also be installed in a small tank connected to the process tank below the liquid level. Hence, the feed should be under slight pressure. Alternatively, the feed may be introduced at the desired flow using a metering pump.

The ZW-1 module stands vertically in the tank, slightly off-center and is fastened with a clamp to the support bracket. It is important to orient the module properly so the air is uniformly distributed throughout the membrane. Therefore, after installation, fill the tank with tap water and turn on the air and examine air distribution visually.

ZW MODULE

The module dimensions are shown in Figure 2. A ZW-1 module comes with an aeration tube that is also used to attach the module to the support bracket to hold it in place vertically. It has one hole on top header, both for permeate flow and pressure measurement. The permeate is drawn only from the top header. The center aeration tube supplies air close to the bottom header where orifices are located.

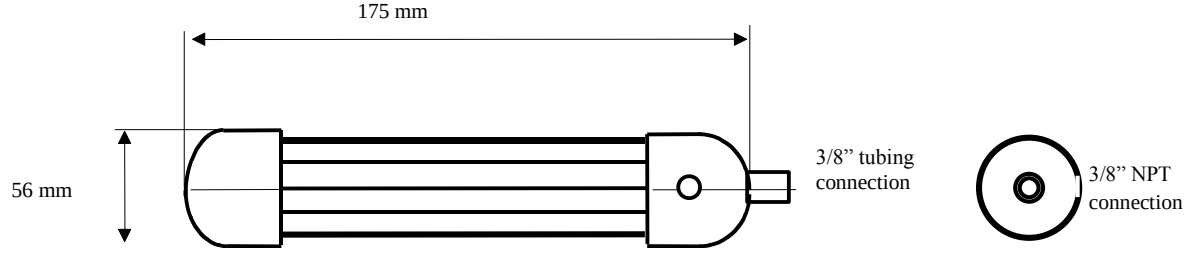

Fig 2. ZW-1 Dimensions (side and top view)

AIR SUPPLY

Air is supplied for module scouring and, if required, biological oxidation (as in ZenoGem applications). The air can be supplied from a convenient compressed air line (oil-free) or by an oil-free blower/air pump. The air from the compressed air line/blower goes through a flow control valve V1 and a rotameter FI en route to the module. 3/8" polyethylene tubing is used for air transmission.

BACKPULSE TANK (OPTIONAL)

The backpulse tank is a 1 liter polyethylene beaker or graduated cylinder. This tank is customer supplied. It is important that the permeate be introduced near the bottom of this tank to avoid air entrainment when the flow is reversed.

SYSTEM SPECIFICATIONS

ZW-1 MODULE SPECIFICATIONS

Model	ZW-1, Submersible Module
Configuration	Outside/In Hollow Fiber
Nominal Membrane Surface Area	0.047 m^2
Membrane Type	Zenon Proprietary
Permeate (Fiber Side) Hold-up Volume	10 mL)

ZW-1 SYSTEM RECOMMENDATIONS

Designation	ZW-1 Bench Test Unit
Electrical service required	115/230 V, 50/60 Hz, 2A
Process tank total volume	2 to 20 L
Process tank working volume, nominal	2 to 20 L
Backpulse tank total volume	1 L
Backpulse tank working volume	1 L
Nominal Permeate flow at 20 LMH	15 millilitres/minute

OPERATING LIMITS

Recommended pump capacity	0 to 150 milliliters/minute
Maximum Transmembrane Pressure	62 kPa (9.0 psig)
Typical Operating TMP	10-50 kPa (1.0-7.0 psi)
Maximum Operating Temperature	40°C (104°F)
Operating pH range	5-9
Maximum Cleaning Temperature	40°C (104°F)
Cleaning pH Range	2-10.5
Maximum OCl^- Exposure (see note)	1000 mg/L
Maximum TMP Back Wash Pressure	55 kPa (8.0 psi)
Maximum Aeration Flow per Module	1.8 m^3/h (1 scfm)

Note: Higher OCl^- concentrations can occasionally be used. Contact Zenon Membrane Products.

SYSTEM ASSEMBLY AND STARTUP

SYSTEM ASSEMBLY

Please read the instructions through completely before proceeding:

1. Acquire auxiliary components and familiarize yourself with them. Note: do not remove ZW-1 module from package. It will remain sealed until startup.
2. Identify a suitable location for the tank, process pump and blower. The drain valve should be near an area floor drain. The unit should be on a table or shelf.
3. Install battery in pressure transmitter. Note, if this unit has been previously used, battery may already be installed. Test pressure transmitter (on/off switch on back). Turn off until needed.
4. Assemble tubing according to drawings in Figure 1. Install temperature indicator, and float feed valve on process tank. Install tubing. Do not connect to ZW-1 module (it is still in package). Note: feed flow should be turned off upstream of float valve. This valve will only be opened when system is ready for operation on process water.
5. Connect pump to permeate tubing.
6. Connect blower to air tubing.
7. Ensure that all equipment switches are in off position, then plug power cords into suitable receptacle.
8. Set V1 to ½ open. Test blower by turning on. Adjust V1 until air flow is 1 scfm (1.8 m3/h). Check for air leaks at connections with dilute liquid soap or similar product. Turn off blower when flow is confirmed; do not close V1.
9. Test process pump operation by simply drawing water from process tank, fill up backpulse tank and let it overflow to process tank. Check for vacuum leaks in tubing. Understand the operation of the pump and learn how to adjust flow direction and motor speed.

SYSTEM STARTUP

1. Unpack ZW-1 module and spray module thoroughly with clean warm (maximum 40°C) water.
2. Fasten ZW-1 module to support bracket with clamp. Set height so liquid level is 2 – 5 cm above top header (with float valve just closed). Connect air, permeate and pressure indicator tubing to module.
3. Turn on air and pump and confirm flowrates. Check for leaks. Adjust airflow to a maximum of 1 scfm if required. In applications with low suspended solids,

substantially lower air flow will be required and would need to be determined individually for each case. For process fluids that are potentially hazardous, lower air flow should be used to prevent mist formation, and the unit should be placed in a fume hood to ensure safety. Adjust module orientation if required to maximize air flow through the fibre bundle. Turn the air off..

4. Direct permeate to drain at 50 mL/min for five minutes .
5. Drain the process tank.
6. Refill process tank with potable water; add sufficient NaOCl to produce a 200 ppm solution in the process tank.
7. Soak the module in 200 ppm NaOCl solution at room temperature for a minimum of 5 hours. Dump process tank contents and rinse; refill with potable water.
8. Start system. Determine clean water permeability: measure flux at 35 kPa (5 psi) transmembrane pressure (TMP) to calculate permeability as lmh/bar. Save this value for future use to determine cleaning efficiency.
9. Unit is ready for testing on process fluid and run can now be started.
10. Measure flow rate, temperature and TMP immediately and periodically thereafter.

Important Notes:

1. A new module is wetted with glycerin for preservation. Do not open the package until ready to use.
2. **Membrane must not dry out**. For short-term storage, keep the membrane under water.

OPERATING INSTRUCTIONS

ROUTINE OPERATION

The ZW-1 unit is intended to operate at a fixed flux (permeate flow) without continuous supervision. Running at a fixed pressure requires constant attention and adjustment of permeate flow. The unit should be periodically inspected to confirm that it is running properly, i.e. tank level is at the float level, the pump is permeating, etc. Whenever data is not being read, the pressure indicator should be turned off to maximize battery life.

The unit can be operated in several modes:

a. Batch Mode

In batch mode a fixed volume of feed is concentrated to the desired volume by filtering through the module and discharging the permeate. The concentrate and permeate are periodically analysed for quality and the operation parameters of temperature, pressure and flow are periodically measured.

b. Semi-batch Mode (fill and draw)

In semi-batch mode the system is run in recirculation mode, ie. the permeate is recycled back to the process tank. Periodically a portion of the permeate is sent to drain. This portion then is made up by adding a corresponding volume new feed to the process tank. The volume and frequency of permeate wasting determine the hydraulic residence time (HRT). The solids residence time (SRT) of the system can also be controlled by periodically wasting to drain a portion of the concentrate and making up with new feed.

c. Continuous Mode

Continuous mode consists of constant permeate flow to drain with continuous or intermittent wasting of the concentrate. This is the most common method of operation with the activated sludge ZenoGem process.

Cleaning:

A soak cleaning will periodically be required. If there is excessive solids build up in the module, or if the permeation TMP reaches 8 psi the module should be removed from the process and placed in a soak tank (2 to 4 litre graduated cylinder). For highly fouling process stream, it is advised to clean daily, using two modules, with one in use while the other being cleaned. The general protocol requires removing any cake apparent on the membranes by hand or with a gentle spray over a drain and soaking the module in 200 ppm

NaOCl at room temperature for a minimum of 5 hours. If there is inorganic fouling, rinse system thoroughly and perform an additional soak in citric acid (5 gm/L) solution for a minimum of 5 hours. Rinse module well with clean water between and after each step.

DATA LOGGING

The following operating data should be recorded regularly during the test:

1. Permeate flowrate
2. Reject or effluent flowrate (daily wasting)
3. Air flowrate
4. Pressure
5. Temperature
6. Accumulated run time
7. All process parameters (e.g. pH, COD, TSS,...)

KEY CONTACTS

If you have any process related questions (e.g. recommended cleaning protocols) or need other general application related information, contact your local Zenon office or technical support staff.

If you have specific questions about the system hardware or if you have suggestions as how to improve the ZW-1 design, contact either your local Zenon office or:

Mr. Henry Behmann.
Corporate Technology
Zenon Environmental Inc.
3239 Dundas St. W., Oakville, Ontario, Canada 6M 4B2
Telephone: (905) 465-3030 (ext. 3094)
Fax: (905) 469-2248
E-mail:hbehmann@zenonenv.com

Appendix A

1. Equipment recommendations (equivalents from other suppliers are acceptable):

Instrumentation

Air Flowmeter (with valve)	- Cole Parmer Catalog # U32460-50
(without)	- Cole Parmer Catalog # U03279-10
Digital Pressure Indicator	- Cole Parmer Catalog # U68925-25
Digital Thermometer	- Cole Parmer Catalog # U90380-00
Stopwatch	- Cole Parmer Catalog # U94410-42

Pumps, permeate / air

Micropump controller	- Cole Parmer Catalog # U75225-00
+ Micropump head	- Cole Parmer Catalog # U07002-25
or	
Masterflex controller	- Cole Parmer Catalog # U77200-00
+ Masterflex pump head	- Cole Parmer Catalog # U77200-62
+ Masterflex tubing	- Cole Parmer Catalog # U06404-14
Diaphragm air pump	- Cole Parmer Catalog # U07061-20

Valve

Air Flow valve	¼" needle valve	- local supplier
Float valve	¼" or 3/8" small float	- local plumbing supplier

Tank

Process tank – 2 litre graduated cylinder to 20 litre pail – plastic preferred

Miscellaneous

¼" OD polyethylene tubing for liquid
3/8" OD polyethylene tubing for air
Plastic compression fittings for above
100 mL graduated cylinder for flow measurement

Vitae

Name	:Waleed Khalid Al-Nowaiser
Nationality	:Saudi
Date of Birth	:8/30/1988
Email	:nowaiserw@gmail.com
Address	:Ibn Abi Al-Sarh Street, Jameyeen, Dammam, Saudi Arabia
Academic Background	:BSc in Civil Engineering, Jubail University College, 2011
Training & Participation	:English Course, University Canada West, 2008
	City Planning Conference, Jubail, 2010
	Jubail First Corrosion Conference, Jubail, 2010